

## SEARCH REQUEST FORM

Scientific and Technical Information Center

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Requester's Full Name: William G. Wright Sr. Examiner #: 62100 Date: 8/20/03  
 Art Unit: 1754 Phone Number 305-7792 Serial Number: 09/997,271  
 Mail Box and Bldg/Room Location: CP3-9415 Results Format Preferred (circle): PAPER DISK E-MAIL

If more than one search is submitted, please prioritize searches in order of need.

\*\*\*\*\*

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Crystallization process for producing highly concentrated hydrogen peroxide  
 Inventors (please provide full names): Stefan Nordhoff; Matthias Greig; Stefan Gross; Rudolf Wagner.

Earliest Priority Filing Date: ? Feb 20, 2001

\*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

*Attorney elected claims 1-19 as group of a restriction. Only look at claims 1-19.*

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## STAFF USE ONLY

	Type of Search	Vendors and cost where applicable
Searcher: <u>EL</u>	NA Sequence (#) _____	STN <u>\$11.41</u>
Searcher Phone #: _____	AA Sequence (#) _____	Dialog _____
Searcher Location: _____	Structure (#) _____	Questel/Orbit _____
Date Searcher Picked Up: _____	Bibliographic <input checked="" type="checkbox"/> _____	Dr.Link _____
Date Completed: <u>8-20-03</u>	Litigation _____	Lexis/Nexis _____
Searcher Prep & Review Time: <u>10</u>	Fulltext _____	Sequence Systems _____
Clerical Prep Time: _____	Patent Family _____	WWW/Internet _____
Online Time: <u>100</u>	Other _____	Other (specify) _____

09/997,271

We claim:

1. A process for the continuous preparation of concentrated hydrogen peroxide of concentration  $c_p$  from aqueous hydrogen peroxide of concentration  $c_E$ ,  $c_E$  being at least 80 wt.% and  $c_p$  being greater than  $c_E$ , comprising:  
suspension crystallizing aqueous hydrogen peroxide of concentration  $c_E$  to obtain hydrogen peroxide crystals and after-treating of the hydrogen peroxide crystals contained in the suspension, wherein the after-treating takes the form of countercurrent washing in a hydraulic or mechanical washing column with a packed crystal bed, and washing said hydrogen peroxide crystals with molten hydrogen peroxide of concentration  $c_p$  as the washing medium.
2. The process according to claim 1, further comprising concentrating aqueous hydrogen peroxide of concentration  $c_E$  in the range from 85 to 95 wt.%, in one step to a concentration  $c_p$  of over 98 wt.%.
3. The process according to claim 1, further comprising concentrating aqueous hydrogen peroxide of concentration  $c_E$  in the range from 88 to 92 wt.%, in one step to a concentration  $c_p$  of equal to or greater than 99.9 wt.%.
4. The process according to claim 1, wherein the suspension crystallizing is carried out in a single- or multi-stage scraper-type crystallizer.
5. The process according to claim 2, wherein the suspension crystallizing is carried out in a single- or multi-stage scraper-type crystallizer.

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6. The process according to claim 3, wherein the suspension crystallizing is carried out in a single- or multi-stage scraper-type crystallizer.
- 5 7. The process according to claim 1, further comprising wherein the suspension crystallizing is carried out in a scraper-type crystallizer, the countercurrent washing of the crystals, combined with solid-liquid separation of the suspension, is carried out in a mechanical washing column, and a portion of the mother liquor that leaves the washing column is fed back into the scraper-type crystallizer.
- 10 8. The process according to claim 2, further comprising wherein the suspension crystallizing is carried out in a scraper-type crystallizer, the countercurrent washing of the crystals, combined with solid-liquid separation of the suspension, is carried out in a mechanical washing column, and a portion of the mother liquor that leaves the washing column is fed back into the scraper-type crystallizer.
- 15 9. The process according to claim 3, further comprising wherein the suspension crystallizing is carried out in a scraper-type crystallizer, the countercurrent washing of the crystals, combined with solid-liquid separation of the suspension, is carried out in a mechanical washing column, and a portion of the mother liquor that leaves the washing column is fed back into the scraper-type crystallizer.
- 20 25 10. The process according to claim 4, further comprising wherein the suspension crystallizing is carried out in a scraper-type crystallizer, the countercurrent washing of the crystals, combined with solid-liquid separation of the suspension, is carried out in a mechanical washing column, and a portion of the
- 30

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mother liquor that leaves the washing column is fed back into the scraper-type crystallizer.

- 5 11. The process according to claim 5, further comprising wherein the suspension crystallizing is carried out in a scraper-type crystallizer, the countercurrent washing of the crystals, combined with solid-liquid separation of the suspension, is carried out in a mechanical washing column, and a portion of the mother liquor that leaves the washing column is fed back into the scraper-type crystallizer.
- 10 12. The process according to claim 4, further comprising nucleation in the suspension crystallizing is carried out at a temperature in the range from greater than 0 to 5 K, below the equilibrium temperature of the  $H_2O_2$  starting material, and the temperature is then lowered further until a suspension density in the range from 5 to 80 % is achieved.
- 15 13. The process according to claim 4, further comprising nucleation in the suspension crystallizing is carried out at a temperature in the range from greater than 0.5 to 3 K, below the equilibrium temperature of the  $H_2O_2$  starting material, and the temperature is then lowered further until a suspension density in the range from 5 to 80 % is achieved.
- 20 14. The process according to claim 7, further comprising nucleation in the suspension crystallizing is carried out at a temperature in the range from greater than 0 to 5 K, below the equilibrium temperature of the  $H_2O_2$  starting material, and the temperature is then lowered further until a suspension density in the range from 5 to 80 % is achieved.
- 25 30

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15. The process according to claim 1, wherein the suspension crystallization is carried out to a suspension density in the range from 20 to 50 %.
- 5 16. The process according to claim 1, wherein the suspension crystallization is carried out to a suspension density in the range from 20 to 30 %.
- 10 17. The process according to claim 1, further comprising hydrogen peroxide having a concentration  $c_p$  of at least 98 wt.%, that leaves the washing column in the form of a melt is stabilized with an effective amount of one or more stabilizers.
- 15 18. The process according to claim 1, further comprising hydrogen peroxide having a concentration  $c_p$  of at least 99 wt.%, that leaves the washing column in the form of a melt is stabilized with an effective amount of one or more stabilizers.
- 20 19. The process according to claim 1, further comprising hydrogen peroxide having a concentration  $c_p$  of at least 99 wt.%, that leaves the washing column in the form of a melt is stabilizing with a stabilizer from the group of tin compounds, phosphates, di- and tri-phosphates, phosphonates and radical acceptors.
- 25 20. Hydrogen peroxide having a concentration of from 99.9 to 100 wt.% and a content of TOC, nitrate, phosphate, nickel and tin of in each case less than 4 mg/l, preferably less than 1 mg of Ni/l, less than 1 mg of Sn/l and less than 2 mg of phosphate/l.
- 30 21. Use of the hydrogen peroxide according to claim 20 as an oxidising agent for motors or for the treatment of electronic components.

09/997,271

# Abstract

A process for the continuous preparation of very highly concentrated hydrogen peroxide having a content of greater than 80 wt.%, especially over 98 wt.%, by  
5 suspension crystallization and after-treatment of the  $H_2O_2$  crystals. The after-treatment takes the form of countercurrent washing in a hydraulic or mechanical washing column with a packed crystal bed. Hydrogen  
10 peroxide is disclosed having a concentration of from 99.9 to 100 wt.% and a content of TOC, nitrate, phosphate, nickel and tin of in each case less than 4 mg/l.

00007271 443004

=> file home

FILE 'HOME' ENTERED AT 18:38:18 ON 20 AUG 2003

=> display history full l1-

FILE 'REGISTRY' ENTERED AT 17:29:00 ON 20 AUG 2003

E HYDROGEN PEROXIDE/CN

L1 1 SEA "HYDROGEN PEROXIDE"/CN

FILE 'LCA' ENTERED AT 17:29:15 ON 20 AUG 2003

L2 32135 SEA (PRODUC? OR PROD# OR GENERAT? OR MANUF? OR MFR# OR  
CREAT? OR FORM## OR FORMING# OR FORMAT? OR MAKE# OR  
MADE# OR MAKING# OR FABRICAT? OR SYNTHESI? OR PREPAR? OR  
PREP#)/BI,AB

FILE 'HCA, WPIDS' ENTERED AT 17:35:14 ON 20 AUG 2003

L3 31391 SEA (L1 OR HYDROGEN#(A) PEROXIDE# OR H2O2) (3A) L2

L4 4991 SEA (L1 OR HYDROGEN#(A) PEROXIDE# OR H2O2) (3A) L2

TOTAL FOR ALL FILES

L5 36382 SEA (L1 OR HYDROGEN#(A) PEROXIDE# OR H2O2) (3A) L2

L6 1805250 SEA CRYST? OR RECRYST?

L7 340029 SEA CRYST? OR RECRYST?

TOTAL FOR ALL FILES

L8 2145279 SEA CRYST? OR RECRYST?

L9 31340 SEA COUNTERCURRENT? OR COUNTER? (2A) CURRENT?

L10 11569 SEA COUNTERCURRENT? OR COUNTER? (2A) CURRENT?

TOTAL FOR ALL FILES

L11 42909 SEA COUNTERCURRENT? OR COUNTER? (2A) CURRENT?

L12 7674 SEA SCRAPE OR SCRAPES OR SCRAPED OR SCRAPING# OR  
SCRAPPER#

L13 22405 SEA SCRAPE OR SCRAPES OR SCRAPED OR SCRAPING# OR  
SCRAPPER#

TOTAL FOR ALL FILES

L14 30079 SEA SCRAPE OR SCRAPES OR SCRAPED OR SCRAPING# OR  
SCRAPPER#

L15 79 SEA (MOLTEN? OR MELT? OR FUSE# OR FUSING# OR FUSION?) (3A)  
(L1 OR HYDROGEN#(A) PEROXIDE# OR H2O2)

L16 27 SEA (MOLTEN? OR MELT? OR FUSE# OR FUSING# OR FUSION?) (3A)  
(L1 OR HYDROGEN#(A) PEROXIDE# OR H2O2)

TOTAL FOR ALL FILES

L17 106 SEA (MOLTEN? OR MELT? OR FUSE# OR FUSING# OR FUSION?) (3A)  
(L1 OR HYDROGEN#(A) PEROXIDE# OR H2O2)

L18 13 SEA L15 AND L3

L19 10 SEA L16 AND L4

TOTAL FOR ALL FILES

L20 23 SEA L17 AND L5

L21 1632 SEA L3 AND L6

L22 243 SEA L4 AND L7

TOTAL FOR ALL FILES

L23 1875 SEA L5 AND L8

L24 39 SEA L3 AND L9

L25 31 SEA L4 AND L10  
TOTAL FOR ALL FILES  
L26 70 SEA L5 AND L11  
L27 8 SEA L3 AND L12  
L28 1 SEA L4 AND L13  
TOTAL FOR ALL FILES  
L29 9 SEA L5 AND L14  
L30 7 SEA L24 AND L21  
L31 1 SEA L25 AND L22  
TOTAL FOR ALL FILES  
L32 8 SEA L26 AND L23  
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L34 27920 SEA L1 OR HYDROGEN#(A) PEROXIDE# OR H2O2  
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L35 193156 SEA L1 OR HYDROGEN#(A) PEROXIDE# OR H2O2  
L36 12428 SEA L33 AND L6  
L37 1468 SEA L34 AND L7  
TOTAL FOR ALL FILES  
L38 13896 SEA L35 AND L8  
L39 270 SEA L33 AND L9  
L40 115 SEA L34 AND L10  
TOTAL FOR ALL FILES  
L41 385 SEA L35 AND L11  
L42 54 SEA L33 AND L12  
L43 18 SEA L34 AND L13  
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L45 79 SEA L33 AND L15  
L46 27 SEA L34 AND L16  
TOTAL FOR ALL FILES  
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L49 4 SEA L37 AND L40  
TOTAL FOR ALL FILES  
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L51 6 SEA L36 AND L42  
L52 1 SEA L37 AND L43  
TOTAL FOR ALL FILES  
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L54 8 SEA L36 AND L45  
L55 5 SEA L37 AND L46  
TOTAL FOR ALL FILES  
L56 13 SEA L38 AND L47  
L57 0 SEA L39 AND L42  
L58 0 SEA L40 AND L43  
TOTAL FOR ALL FILES  
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L60 0 SEA L39 AND L45  
L61 1 SEA L40 AND L46  
TOTAL FOR ALL FILES  
L62 1 SEA L41 AND L47  
L63 0 SEA L42 AND L45



L64 0 SEA L43 AND L46  
TOTAL FOR ALL FILES  
L65 0 SEA L44 AND L47  
  
FILE 'HCA' ENTERED AT 17:51:58 ON 20 AUG 2003  
L66 28 SEA L27 OR L30 OR L51 OR L54  
L67 12 SEA L18 NOT L66  
  
FILE 'WPIDS' ENTERED AT 17:52:45 ON 20 AUG 2003  
L68 10 SEA L28 OR L31 OR L49 OR L52 OR L55 OR L61  
L69 6 SEA L19 NOT L68  
  
FILE 'HCA, WPIDS' ENTERED AT 17:55:51 ON 20 AUG 2003  
L70 2888 SEA SUSPEN?(2A) (CRYST? OR RECRYST?)  
L71 1088 SEA SUSPEN?(2A) (CRYST? OR RECRYST?)  
TOTAL FOR ALL FILES  
L72 3976 SEA SUSPEN?(2A) (CRYST? OR RECRYST?)  
L73 7 SEA L70 AND L3  
L74 5 SEA L71 AND L4  
TOTAL FOR ALL FILES  
L75 12 SEA L72 AND L5  
L76 31 SEA L33 AND L70  
L77 13 SEA L34 AND L71  
TOTAL FOR ALL FILES  
L78 44 SEA L35 AND L72  
  
FILE 'HCA' ENTERED AT 17:59:25 ON 20 AUG 2003  
L79 1 SEA L76 AND (L9 OR L12 OR L15)  
L80 6 SEA (L73 OR L79) NOT (L66 OR L67)  
  
FILE 'WPIDS' ENTERED AT 18:01:46 ON 20 AUG 2003  
L81 4 SEA L74 NOT (L68 OR L69)  
L82 8 SEA L77 NOT (L68 OR L69 OR L81)

=> file wpids

FILE 'WPIDS' ENTERED AT 18:38:33 ON 20 AUG 2003  
COPYRIGHT (C) 2003 THOMSON DERWENT

FILE LAST UPDATED: 20 AUG 2003 <20030820/UP>  
MOST RECENT DERWENT UPDATE: 200353 <200353/DW>  
DERWENT WORLD PATENTS INDEX SUBSCRIBER FILE, COVERS 1963 TO DATE

=> d l68 1-10 max

L68 ANSWER 1 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 2003-504422 [47] WPIDS  
DNN N2003-400571 DNC C2003-134785  
TI Treatment apparatus, for ~~treating exhaust gas~~ containing volatile  
organic compounds, has wet scrubber, oxidation unit, and recycling  
device.

DC J01 L03 U11  
IN LAI, C; LIN, S; WU, H  
PA (LAIC-I) LAI C; (LINS-I) LIN S; (WUHH-I) WU H  
CYC 1  
PI US 2003094099 A1 20030522 (200347)\* 9p B01D053-14  
ADT US 2003094099 A1 US 2002-197811 20020719  
PRAI TW 2001-122838 20010914  
IC ICM B01D053-14  
AB US2003094099 A UPAB: 20030723

NOVELTY - A treatment apparatus, for treating an exhaust gas containing volatile organic compounds, comprises a wet scrubber for receiving the exhaust gas, and transporting organic pollutants in the exhaust gas into a scrubbing water; an oxidation unit for introducing an oxidizing agent comprising ozone into the scrubbing water; and a recycling device for recycling the scrubbing water.

DETAILED DESCRIPTION - A treatment apparatus, for treating an exhaust gas containing volatile organic compounds, comprises a wet scrubber (10) for receiving the exhaust gas, and transporting organic pollutants in the exhaust gas into a scrubbing water; an oxidation unit for receiving the scrubbing water from the wet scrubber, thus introducing an oxidizing agent containing ozone into the scrubbing water to cause oxidation reaction; and a recycling device (8) for recycling the scrubbing water from the oxidation unit to the wet scrubber, so that the scrubbing water absorbs organic pollutants in the exhaust gas.

USE - For treating an exhaust gas containing volatile organic compounds, such as that released from semiconductor or liquid **crystal** display manufacture processes (claimed)

ADVANTAGE - The novel process treats volatile organic compounds (VOCs) exhaust gas with lower operational costs. It treats the VOCs exhaust gas that is not affected by high boiling points substances. It treats the exhaust gas that greatly reduces both the amounts of required clean water and wastewater production. It treats the exhaust gas that exhibits high efficiency of VOC removal and ozone gas use.

DESCRIPTION OF DRAWING(S) - The figure illustrates an oxidation scrubbing system, as above.

Oxidation tanks 6, 7

Recycling device 8

Wet scrubber 10

Monitoring system 19

Additive dispenser 21

Dwg.1/5

TECH US 2003094099 A1UPTX: 20030723

TECHNOLOGY FOCUS - CHEMICAL ENGINEERING - Preferred Component: The scrubber is vertical or horizontal. The contact mode of the exhaust gas and the scrubbing water is **countercurrent** or concurrent. The apparatus further comprises an additive dispenser (21) for introducing an additive into the oxidation unit, to maintain the pH value of scrubbing water at 8-11; an outlet piping for draining the scrubbing water from the apparatus; and a monitoring system (19) for observing the treatment of the exhaust

gas. The oxidation unit comprises oxidation tank(s) with at least an inlet for introducing the oxidizing agent. It is set up by two or more oxidation tanks (6, 7) in series.

Preferred Parameter: The hydraulic retention time of the scrubbing water in each oxidation tank is 2-10 minutes.

Preferred Method: An exhaust gas containing volatile organic compounds is treated by introducing the exhaust gas into the wet scrubber, where organic pollutants in the exhaust gas are absorbed by the scrubbing water; pumping the scrubbing water containing the organic pollutants into the oxidation tank, thus causing oxidation reaction between the organic pollutants and the oxidizing agent containing ozone; and introducing the scrubbing water after the oxidation reaction into the wet scrubber, and repeating the first introducing step and the pumping step.

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Component: The organic pollutants are esters, ethers, aldehydes, alcohols, ketones, or organic acids. The exhaust gas further comprises noxious components having molecular structures including nitrogen and sulfur.

TECHNOLOGY FOCUS - INORGANIC CHEMISTRY - Preferred Component: The oxidizing agent further comprises **hydrogen peroxide**.

FS CPI EPI  
FA AB; GI  
MC CPI: J01-D; J01-E02A3; L03-J; L04-X  
EPI: U11-C15Q

L68 ANSWER 2 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 2002-566584 [60] WPIDS

DNC C2002-160553

TI Continuous **preparation of hydrogen peroxide** for e.g., motors, comprises suspension **crystallization** of aqueous **hydrogen peroxide**, and after-treatment of **formed hydrogen peroxide crystals**.

DC E36 L03

IN CREUTZ, M; GROSS, S; NORDHOFF, S; WAGNER, R

PA (DEGS) DEGUSSA AG; (CREU-I) CREUTZ M; (GROS-I) GROSS S; (NORD-I) NORDHOFF S; (WAGN-I) WAGNER R

CYC 38

PI WO 2002044083 A1 20020606 (200260)\* EN 18p C01B015-013

RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE TR

W: AU BR CA IL JP MX NO NZ PL SG SI ZA

EP 1213262 A1 20020612 (200260) DE C01B015-013

R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK  
NL PT RO SE SI TR

US 2002068035 A1 20020606 (200260) C01B015-01

AU 2002021739 A 20020611 (200264) C01B015-013

ADT WO 2002044083 A1 WO 2001-EP12293 20011024; EP 1213262 A1 EP

2000-126387 20001202; US 2002068035 A1 US 2001-997271 20011130; AU

*applicants  
pub.*

2002021739 A AU 2002-21739 20011024  
FDT AU 2002021739 A Based on WO 200244083  
PRAI EP 2000-126387 20001202  
IC ICM C01B015-01; C01B015-013  
AB WO 200244083 A UPAB: 20021031

NOVELTY - Concentrated **hydrogen peroxide** is continuously **prepared** by suspension **crystallization** of aqueous **hydrogen peroxide** of first concentration (80 wt.%). Produced **hydrogen peroxide crystals** in the suspension are after-treated by **countercurrent** washing in hydraulic or mechanical washing column with a packed **crystal bed** using a **produced molten hydrogen peroxide** as washing medium.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for a **hydrogen peroxide** having concentration of 99.9-100 wt.%, and content of total organic carbon (TOC), nitrate, phosphate, nickel (Ni), and tin (Sn). The **hydrogen peroxide** comprises less than 4 mg TOC/l, preferably less than 1 mg Ni/l, less than 1 mg Sn/l, and less than 2 mg phosphate/l.

USE - For continuously **preparing hydrogen peroxide** for motors or for the treatment of electronic components (claimed).

ADVANTAGE - The inventive process **produces hydrogen peroxide** of very high concentration i.e., over 98 (preferably 99.9) wt.% and lower content of impurities, e.g., organic carbon and conventional stabilizers.

DESCRIPTION OF DRAWING(S) - The drawing shows a process diagram.

Mechanical washing column 5

Dwg.1/1

TECH WO 200244083 A1UPTX: 20020919

TECHNOLOGY FOCUS - CHEMICAL ENGINEERING - Preferred Process: The suspension **crystallization** is carried out in the single- or multi-stage scraper-type **crystallizer** to reach a suspension density of 20-50 (preferably 20-30)%. The **countercurrent** washing of the **crystals**, combined with solid-liquid separation of the suspension, is carried out in mechanical washing column (5). A portion of the mother liquor that leaves the washing column is fed back into the scraper type **crystallizer**. Nucleation in the suspension **crystallization** is carried out at 0-5 (preferably 0.5-3)degreesK, below the equilibrium temperature of the **hydrogen peroxide** starting material having the first concentration of 85-95 (preferably 88-92)%. The temperature is then lowered further until a suspension density of 5-80% is achieved.

**Hydrogen peroxide** with a concentration of 98 (preferably 99.9) wt.% that leaves the washing column in the form of a melt is stabilized by stabilizers.

TECHNOLOGY FOCUS - INORGANIC CHEMISTRY - Preferred Stabilizers: The

stabilizers are selected from the group of tin compounds, phosphates, di- and tri-phosphates, phosphonates and radical acceptors.

KW [1] 209-0-0-0 CL PRD  
 FS CPI  
 FA AB; GI; DCN  
 MC CPI: E11-Q01; E31-E; L03-B02F  
 DRN 1732-P; 1732-U  
 CMC UPB 20021031  
 M3 \*01\* C101 C408 C550 C730 C800 C801 C802 C804 C805 C807 M411 M424  
 M720 M904 M905 M910 N164 N511 Q020 Q454 R032  
 DCN: R01732-K; R01732-P

L68 ANSWER 3 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
 AN 2002-395860 [43] WPIDS  
 DNC C2002-111584

TI Concentration of aqueous ~~hydrogen peroxide~~ to product useful as high energy source involves batch **crystallization** by wetting surface with solution and cooling to form seed **crystals**, adding supercooled educt and sweating **crystals** formed..

DC E36  
 IN CREUTZ, M; GLENNEBERG, J; MOELLER, R; NORDHOFF, S; WAGNER, R; MOLLER, D; MOELLER, D

PA (DEGS) DEGUSSA AG; (CREU-I) CREUTZ M; (GLEN-I) GLENNEBERG J; (MOLL-I) MOLLER D; (NORD-I) NORDHOFF S; (WAGN-I) WAGNER R

CYC 28

PI DE 10054742 A1 20020508 (200243)\* 5p C01B015-013

US 2002062536 A1 20020530 (200244) C01B015-13

WO 2002036487 A1 20020510 (200244) EN C01B015-013

RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE TR

W: CA IL JP MX NO NZ SG ZA

NO 2003001991 A 20030502 (200349) C01B015-013

ADT DE 10054742 A1 DE 2000-10054742 20001104; US 2002062536 A1 US 2001-983995 20011026; WO 2002036487 A1 WO 2001-EP11460 20011004; NO 2003001991 A WO 2001-EP11460 20011004, NO 2003-1991 20030502

PRAI DE 2000-10054742 20001104

IC ICM C01B015-013; C01B015-13

AB DE 10054742 A UPAB: 20020709

NOVELTY - Concentration of an aqueous **hydrogen peroxide** (H2O2 educt) to recover the H2O2

product of concentration at least 90 wt.% by discontinuous **crystallization** comprises wetting the cooling surface of a **crystallization** vessel with H2O2 of concentration

not less than 70 wt.% and cooling to form seed **crystals**;

adding educt cooled below the melting point of the seeds and cooling to grow **crystals**; removing uncrystallized H2O2;

and sweating to give product **crystals**.

DETAILED DESCRIPTION - Concentration of an aqueous **hydrogen peroxide** of concentration cE (

H2O2 educt) to recover the H2O2 product of

concentration cP at least 90 wt.% involves discontinuous

*reductant*  
*OPPT*

**crystallization.** The cooling surface of a **crystallization** vessel is (partly) wetted with **H2O2** of concentration not less than 70 wt.%, especially in the cE to cP range, and seed **crystals** are formed on this by cooling to a temperature close to the **melting** point of this **H2O2** at a rate not less than 1 K/second, with a cooling medium on the back of the surface. The vessel is then filled with educt with a temperature below the melting point of the seed **crystals** and the temperature of the cooling medium is reduced to a value in the range below the melting point of the product and -50 deg. C. During and, if necessary, after cooling, **crystals** are allowed to grow from the seed **crystals**. Then uncrystallized **H2O2** is separated and the **crystal** layer is subjected to a sweating process by increasing the temperature to a value in the range from 10 K below to 5 K above the melting point of the product in 0.2-20 hours. The **H2O2** is then separated, giving **crystalline H2O2** of concentration cP.

USE - The high energy content of highly concentrated (90-100 wt.%) **hydrogen peroxide** makes it useful in technology.

ADVANTAGE - Distillation is suitable for recovering aqueous **hydrogen peroxide (H2O2)** solutions with a concentration up to about 90 wt.% but, for cost and safety reasons, not higher concentrations. The strong supercooling tendency of highly concentrated aqueous **H2O2** solutions make concentration by **crystallization** difficult. Fractional batch **crystallization** is inefficient, as the concentration is increased by only 2-4% in each stage. In the present process, a **H2O2** educt with a concentration of 90 plus or minus 2 wt.% is converted to a **H2O2** product with a concentration of 97-99 wt.% in one stage (claimed). The process is simple and can be operated batchwise by personnel with little training. It can be carried out in simple apparatus with low control cost and is also suitable for simple and reliable scale-up to meet increasing demand.

DESCRIPTION OF DRAWING(S) - The drawing shows preferred apparatus for operating the process.

Reservoir for **hydrogen peroxide (H2O2)** educt 1

**Crystallization** vessel 2

Cooling finger 3

Cooling machine 4

Product container 5

Mother liquor container 6

Coolant feed pipe 7

Coolant return pipe 8

Working line for temperature control 9

Drain pipe to product container 10

Educt feed pipe 11

Drain pipe to mother liquor container 12

Temperature gauges T1, T2, TC3

Dwg. 1/4

TECH DE 10054742 A1 UPTX: 20020709

TECHNOLOGY FOCUS - CHEMICAL ENGINEERING - Preferred Process: The cooling surface is a cooling finger, dipping into the **crystallization** vessel, with cooling medium circulating inside the finger; or the **crystallization** vessel is a plate-type heat exchanger. The cooling rate is not less than 5 (10-30) K/second for seed **crystal** formation. The vessel is filled with **H2O2** educt with a temperature between -5 and less than -20degreesC, then the temperature is reduced to -20 to -40degreesC in 0.5-10 hours to form the **crystal** layer. Sweating is carried out by increasing the temperature of the cooling medium to a value in the range from 5 K below to 5 K above (5-1 K below) the melting point of the product in 1-10 hours, so that a **H2O2**-enriched fraction remains in the **crystal** layer and less concentrated **H2O2** drips off as melt

KW [1] 209-0-0-0 CL PRD

FS CPI

FA AB; GI; DCN

MC CPI: E11-R01B; E31-E

DRN 1732-P; 1732-U

CMC UPB 20020709

M3 \*01\* C101 C408 C550 C730 C800 C801 C802 C804 C805 C807 M411 M720  
M904 M905 M910 N104 N164 N511 R032  
DCN: R01732-K; R01732-P

L68 ANSWER 4 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 2001-211309 [21] WPIDS

DNC C2001-062875

TI Production of beet sugar involves macerating beets or beets pieces, mechanically separating juice from the macerated beets, and membrane filtering the separated juice.

DC D17

IN DONOVAN, M; HLAVACEK, M; JANSEN, R P; MANNAPPERUMA, J D; REISIG, R C; WALKER, G; WILLIAMS, J C

PA (TATL) TATE & LYLE INC; (TATL) TATE & LYLE IND LTD; (TATL) TATE & LYLE SUGAR HOLDINGS INC

CYC 95

PI WO 2001014594 A2 20010301 (200121)\* EN 57p C13D000-00

RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC  
MW MZ NL OA PT SD SE SL SZ TZ UG ZW

W: AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CR CU CZ DE  
DK DM DZ EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG  
KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ  
PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG UZ VN YU  
ZA ZW

AU 2000069063 A 20010319 (200136) C13D000-00

US 2001054420 A1 20011227 (200206) C13D003-16

EP 1204767 A2 20020515 (200239) EN C13D003-16

R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK  
NL PT RO SE SI

US 6387186 B1 20020514 (200239) C13D001-00

US 6406547 B1 20020618 (200244) C13D003-16  
 US 6440222 B1 20020827 (200259) C13D001-08  
 SK 2002000377 A3 20021106 (200281) C13D003-16  
 CZ 2002000570 A3 20021113 (200282) C13D001-00  
 HU 2002004353 A2 20030528 (200341) C13D003-16  
 ADT WO 2001014594 A2 WO 2000-US22301 20000815; AU 2000069063 A AU  
 2000-69063 20000815; US 2001054420 A1 Cont of US 1999-376026  
 19990819, US 2001-883400 20010618; EP 1204767 A2 EP 2000-957444  
 20000815, WO 2000-US22301 20000815; US 6387186 B1 US 1999-376026  
 19990819; US 6406547 B1 US 2000-618416 20000718; US 6440222 B1 US  
 2000-618831 20000718; SK 2002000377 A3 WO 2000-US22301 20000815, SK  
 2002-377 20000815; CZ 2002000570 A3 WO 2000-US22301 20000815, CZ  
 2002-570 20000815; HU 2002004353 A2 WO 2000-US22301 20000815, HU  
 2002-4353 20000815  
 FDT AU 2000069063 A Based on WO 200114594; EP 1204767 A2 Based on WO  
 200114594; SK 2002000377 A3 Based on WO 200114594; CZ 2002000570 A3  
 Based on WO 200114594; HU 2002004353 A2 Based on WO 200114594  
 PRAI US 2000-618831 20000718; US 1999-376026 19990819; US 2000-618416  
 20000718; US 2001-883400 20010618  
 IC ICM C13D000-00; C13D001-00; C13D001-08; C13D003-16  
 ICS C13D001-02; C13D003-00; C13D003-08; C13D003-10; C13D003-14  
 AB WO 200114594 A UPAB: 20010418

NOVELTY - Beet sugar is produced by macerating beets or beets pieces, mechanically separating juice from the macerated beets, and membrane filtering the separated juice to produce a retentate and a permeate.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for a process for purifying a sucrose-containing juice obtained from sugar beets comprising introducing air into the juice to cause polymerization of color bodies, and removing some of the color bodies from the juice by membrane filtration, e.g. ultrafiltration membrane and/or nanofiltration membrane.

USE - For producing beet sugar.

ADVANTAGE - The method eliminates lime and carbon dioxide, and produces beet sugar having an ash concentration of not more than 1.0-2.5 wt.% on a dry solid basis (claimed). It produces beet juice of lower color which allows for less washing of the final **crystalline** product. It has lower retention time, reduces the microbial destruction of sucrose, reduces the percentage of sucrose retained in the pulp, and increases the total sugar recovery.

Dwg.0/6

TECH WO 200114594 A2UPTX: 20010418

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Method: The beets are cut into pieces and macerated in an attrition mill. The juice is separated from beets by moving porous vacuum filtration belt with **countercurrent** flow of macerated beets and water, or by centrifugation. The membrane filtration is done at at least 80degreesC, and subjected to diafiltration to recover residual sugar in the retentate. The permeate from the membrane filtration is concentrated by reverse osmosis, evaporated, and **crystallized** to produce sucrose. The separated juice is



filtered through a first ultrafiltration membrane having a molecular weight cutoff of 2000-200000 daltons and a pore size of not more than 0.1  $\mu\text{m}$ , and a second ultrafiltration membrane having a molecular weight cutoff of 500-5000 daltons. The nanofiltration retentate is purified by electrodialysis, softened by ion exchange to remove at least 65 wt.% calcium, magnesium, potassium, sodium, and their inorganic and organic anions. The method further comprises introducing **hydrogen peroxide** and/or ozone into the feed juice prior to filtration; removing at least 90 wt.% residual beet fibers and silt, having a dimension of greater than 50-150  $\mu\text{m}$ , from the separated liquid by screening or filtration prior to filtration; evaporating the nanofiltration retentate to produce concentrated syrup; **crystallizing** white sugar from the concentrated syrup; removing 75% of the raffinose contained in the mother liquor in a simulated bed chromatographic separator; and recycling the mother liquor to one of the ultrafiltration membranes. The recycled liquor is subjected to further purification, evaporation, and **crystallization**. Preferred Components: The pulp still contains not more than 3-5% sucrose after the liquid is separated. The feed juice is at 60-93 (preferably 71-85) degrees C during filtration. The first ultrafiltration permeate has a color of 3000-10000 icu, and the second ultrafiltration permeate has a color of not more than 2500-4000 icu. The nanofiltration permeate comprises (wt.%) ash (at least 30), invert sugars (at least 30), and betaine (at least 25) present in the feed juice.

TECHNOLOGY FOCUS - INORGANIC CHEMISTRY - Preferred Component: The separated juice is pH adjusted to 6-8 by addition of sodium hydroxide; and contacted with an agent from sulfur dioxide, sulfate salts, sulfite salts, and/or bisulfite salts.

FS CPI  
 FA AB  
 MC CPI: D06-A; D06-B; D06-D  
 DRN 0135-P; 1066-P; 1514-U; 1674-U; 1732-U; 1887-U

L68 ANSWER 5 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
 AN 2000-475636 [41] WPIDS  
 DNN N2000-354863 DNC C2000-142519  
 TI Disposable applicators, used to dispense bioactive materials and adhesives to biological sites, and industrial and home application materials, include e.g. frangible ampoule, flexible applicator body..

DC A96 B07 P34  
 IN BADEJO, I T; BAREFOOT, J B; COTTER, W M; D'ALESSIO, K R; HEDGPETH, D L; MAINWARING, L H; NARANG, U; SHERBONDY, A; SZABO, G N  
 PA (CLOS-N) CLOSURE MEDICAL CORP  
 CYC 85  
 PI WO 2000038777 A1 20000706 (200041)\* EN 60p A61M035-00  
 RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC  
 MW NL OA PT SD SE SL SZ TZ UG ZW  
 W: AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI  
 GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR

LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI  
SK SL TJ TM TR TT UA UG UZ VN YU ZW

AU 2000023764 A 20000731 (200050) A61M035-00  
US 6283933 B1 20010904 (200154) A61M035-00  
ADT WO 2000038777 A1 WO 1999-US30542 19991222; AU 2000023764 A AU  
2000-23764 19991222; US 6283933 B1 US 1998-219851 19981223  
FDT AU 2000023764 A Based on WO 200038777  
PRAI US 1999-430290 19991029; US 1998-219851 19981223  
IC ICM A61M035-00  
ICS A46B011-00; A61P001-02; B43K005-12  
AB WO 200038777 A UPAB: 20000831  
NOVELTY - Disposable applicators for dispensing and applying  
materials.

DETAILED DESCRIPTION - Disposable applicators for dispensing  
and applying materials comprise:

(a) a frangible ampoule containing a predetermined quantity of  
dispensable material with a 1st closed end and a 2nd frangibly  
sealed end;

(b) a flexible applicator body with a hollow main applicator  
body portion with a sealed proximal end and a secondary applicator  
body portion with an open distal end, the main applicator body  
portion being of a length and circumference sized to receive (a),  
and the second frangibly sealed end of the ampoule being positioned  
facing the secondary applicator body portion of the applicator;

(c) retaining means for retaining the ampoule substantially  
within the applicator body;

(d) a 1st swab attached to the proximal end of the main  
applicator body portion; and

(e) a 2nd swab attached to and in fluid communication with the  
distal end of the secondary applicator body portion; (a) being  
frangible at the frangibly sealed end by application of a sufficient  
force applied to the ampoule through the applicator body to allow  
flow of the dispensable material toward the 2nd swab.

INDEPENDENT CLAIMS are also included for:

- (1) methods of making disposable applicators;
- (2) methods of treating tissue; and
- (3) kits comprising saleable packages containing disposable  
applicators.

USE - The applicators are used to treat tissues (claimed). The  
applicators are used to apply therapeutic or otherwise biomedically  
useful liquid compositions to surfaces such as biological tissues.  
They may be used to deliver bioactive materials to biological sites  
including medicaments, such as antibiotics, antimicrobials,  
antiseptics, bacteriocins, bacteriostats, disinfectants, steroids,  
anesthetics, antifungals, antiinflammatories, antibacterials,  
antivirals, anti-tumor agents, growth promoters and/or wound-healing  
promoters (quaternary ammonium halides such as benzalkonium chloride  
and benzethonium chloride, chlorhexidine sulfate, gentamicin  
sulfate, **hydrogen peroxide**, quinolone thioureas,  
silver salts such as silver acetate, silver benzoate, silver  
carbonate, silver chloride, silver citrate, silver iodide, silver  
nitrate and silver sulfate, copper compounds, such as copper

chloride, copper sulfate and copper peptides, sodium hypochlorite, sulfadiazine salts including silver, sodium and zinc salts, and/or antioxidants such as vitamins e.g. vitamin E, and adhesives for absorbable and non-absorbable biomedical applications such as tissue adhesives, sealants for preventing bleeding or for covering open wounds, apposing surgically incised or traumatically lacerated tissues, retarding blood flow from wounds, drug delivery, dressing burns, dressing skin or other superficial or surface wounds (abrasions, chaffed or raw skin and/or stomatitis), hernia repair, meniscus repair, and aiding repair and regrowth of living tissue, and in industrial and home applications such as in bonding rubbers, plastics, wood, composites, fabrics and other natural and synthetic materials. They may be used to treat tissues to promote wound healing of leg ulcers and thermal burns, and to apply compositions to stomatitis lesions including inflammation of mucous tissue of the oral cavity such as lesions and sores as well as to skin wounds such as minor cuts, **scrapes**, irritations, compromised skin, superficial lacerations, burns or abrasions, or sores on mucous membranes.

ADVANTAGE - The applicators can be used by a person, such as a patient, to apply biomedically useful compositions conveniently, inexpensively and effectively. They are designed for simple and effective delivery of liquid compositions and for single-handed use by a person with average strength, and require little or no instruction prior to use. The applicators are designed to be simple in construction and use, have no moving parts and require no special disposal procedures. Application of biomedically useful compositions within the applicators does not need to be supervised by a medical professional, but can be performed by the user in an environment and at a time chosen by the user.

DESCRIPTION OF DRAWING(S) - Top view of the applicator.

main body portion 24  
secondary body portion 26  
frangible ampoule 30  
applicator swab 50  
dry wiping swab 60

Dwg.10/11

TECH WO 200038777 A1UPTX: 20000831

TECHNOLOGY FOCUS - PHARMACEUTICALS - Preferred Applicator: (a) is formed from a plastic material, preferably polyethylene terephthalate. The ampoule is frangible by an activation force of less than about 4.3 kg. (b) is formed from a plastic material, preferably a low-density polyethylene, and is especially transparent or translucent. The main applicator body portion has a substantially constant cross-section prior to sealing of the proximal end and the secondary applicator body portion is tapered. (d) is a dry swab formed from an absorbent material, preferably a thermoplastic polyurethane foam, and especially includes a flavorant, bioactive material and/or polymerization or crosslinking initiator, accelerator and/or inhibitor. (e) is formed from an absorbent material, preferably a thermoplastic polyurethane foam, especially which is saturable with the dispensable material and more especially

changes color upon saturation with the dispensable material. (e) is formed of a reticulated material, preferably containing a basic agent, especially caustic soda, hydroxides of light metals, ammonium hydroxide, caustic alcohol and/or silver nitrate. (e) further includes a polymerization or crosslinking initiator, accelerator or inhibitor. (e) includes a flavorant and/or a bioactive material. The bioactive material is **crystal** violet present in an amount sufficient to provide effective antiviral, antimicrobial and/or antifungal properties to a polymerized adhesive composition. The **crystal** violet is present in an amount sufficient to initiate polymerization of a monomeric adhesive composition without providing effective antiviral, antimicrobial and/or antifungal properties to the adhesive composition subsequent to polymerization. The flavorant is 5-fold orange oil, anethole, banana distillate, benzaldehyde, clove oil, cold pressed Valencia orange oil, cold pressed grapefruit oil, cold pressed lemon oil, cold pressed lime oil, cucumber distillate, honey distillate, menthol, alkyl salicylates, monosodium glutamate, spearmint, wintergreen, cinnamon, citrus, cherry, apple, peppermint, peppermint oil, peppermint spirit, vanillin, thymol and/or ethyl vanillin. A size of (e) correlates to an amount of the dispensable material to be applied by (e). (d) and (e) are of different colors. The dispensable material is an alpha-cyanoacrylate adhesive composition, preferably containing n-butyl cyanoacrylate and/or 2-octyl cyanoacrylate. The dispensable material further comprises a medicament. The applicator is covered by a wrapper.

FS CPI GMPI  
 FA AB; GI  
 MC CPI: A12-P06; A12-V03D; B04-C03; B11-C04  
 PLE UPA 20000831

[1.1] 018; H0000; G0420 G0339 G0260 G0022 D01 D12 D10 D26 D51  
 D53 D58 D63 F12 F41 F89 D11 D88; P0088

[1.2] 018; H0000; G0420 G0339 G0260 G0022 D01 D12 D10 D26 D51  
 D53 D58 D63 F12 F41 F89 D11 D92; P0088

[1.3] 018; H0022 H0011; G0420 G0339 G0260 G0022 D01 D12 D10 D26  
 D51 D53 D58 D63 F12 F41 F89 D11 D88; G0420 G0339 G0260  
 G0022 D01 D12 D10 D26 D51 D53 D58 D63 F12 F41 F89 D11 D92;  
 P0088

[1.4] 018; Q9999 Q6644-R; N9999 N7012; Q9999 Q7272; Q9999 Q8037  
 Q7987

[1.5] 018; ND01; K9416; Q9999 Q8026 Q7987

[2.1] 018; P0884 P1978 P0839 H0293 F41 D01 D11 D10 D19 D18 D31  
 D50 D63 D90 E21 E00

[2.2] 018; Q9999 Q8399-R Q8366; B9999 B3930-R B3838 B3747; B9999  
 B4193 B4091 B3838 B3747

[2.3] 018; ND01; K9416; Q9999 Q8026 Q7987

[3.1] 018; H0317; P1592-R F77 D01; S9999 S1309-R

[3.2] 018; Q9999 Q8571 Q8366; Q9999 Q8388 Q8366; B9999 B3383-R  
 B3372

[3.3] 018; ND01; K9416; Q9999 Q8026 Q7987

[4.1] 018; H0124-R

[4.2] 018; N9999 N5721-R

CMC UPB 20000831

M6 \*01\* M905 R112 R120 R130 R150 R301 R420 R501

L68 ANSWER 6 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1999-180049 [15] WPIDS

CR 1992-331449 [40]; 1993-196705 [24]; 1993-288094 [36]; 1995-373524 [48]; 1996-077331 [08]; 1996-077341 [08]; 1996-116793 [12]; 1996-160150 [16]; 1996-209238 [21]; 1996-259569 [26]; 1996-286926 [29]; 1997-020943 [02]; 1997-020944 [02]; 1997-033947 [03]; 1997-033948 [03]; 1997-318573 [27]; 1997-392980 [36]; 1998-031237 [03]; 2000-012268 [54]

DNC C1999-052419

TI Permeation-enhanced wound healing composition - comprises a permeation-enhancing agent and a wound healing composition containing pyruvic acid, an antioxidant, and a mixture of fatty acids.

DC B05

IN MARTIN, A

PA (WARN) WARNER LAMBERT CO

CYC 1

PI US 5874479 A 19990223 (199915)\* 40p A61K031-045

ADT US 5874479 A Cont of US 1991-663500 19910301, CIP of US 1993-53922 19930426, CIP of US 1994-224936 19940408, US 1998-19457 19980205

PRAI US 1998-19457 19980205; US 1991-663500 19910301; US 1993-53922 19930426; US 1994-224936 19940408

IC ICM A61K031-045

ICS A61K031-07; A61K031-355

AB US 5874479 A UPAB: 20000606

Permeation-enhanced wound healing composition comprises a permeation-enhancing agent (I) and a wound healing composition (II). (II) comprises: (a) pyruvic acid and/or its salts; (b) an antioxidant; and (c) a mixture of optionally saturated fatty acids, which are required for the resuscitation of injured mammalian cells. Components (a), (b) and (c) have a synergistic effect.

USE - The composition is useful for the treatment of wounds (claimed). The composition can also be used for moisturising and protecting skin, healing dry cracked skin, treating irritated skin (e.g. diaper rash), healing severe dry skin due to other diseases (e.g. venous dermatitis), treating psoriasis and other hyper-proliferative diseases, protecting skin from UV light damage (antioxidant skin replacement), treating seborrheic conditions, and treating shaving wounds (in the form of an after shave lotion). Other uses include healing of: cuts and **scrapes**; burns; decubitus ulcers; bed sores; fissures and haemorrhoids; post surgical wounds; diabetic ulcers; and venous ulceration.

ADVANTAGE - The composition has the ability to simultaneously decrease cellular levels of **hydrogen peroxide production**, increase cellular resistance to cytotoxic agents, increases rates of cellular proliferation, increase cellular viability to protect and resuscitate mammalian cells, and enhance penetration of actives into membranes.

Dwg.0/8

FS CPI  
FA AB; DCN

L68 ANSWER 7 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1999-005662 [01] WPIDS

DNN N1999-004503 DNC C1999-002143

TI **Hydrogen peroxide** solution manufacturing apparatus for electronic devices - has an ion exchange membrane arranged between anode and cathode as solid electrolyte.

DC E36 J03 L03 U11 U14

PA (KATA-N) KATAKURA KAGAKU KOGYO KENKYUSHO KK; (PERE) PERMELEC ELECTRODE LTD

CYC 1

PI JP 10280180 A 19981020 (199901)\* 6p C25B001-30

ADT JP 10280180 A JP 1997-99735 19970402

PRAI JP 1997-99735 19970402

IC ICM C25B001-30

ICS C01B015-01

AB JP 10280180 A UPAB: 19990107

NOVELTY - An ion exchange membrane which functions as solid electrolyte is installed between anode and cathode. DETAILED DESCRIPTION - The apparatus has a reaction chamber in which a cathode containing hydrogen occlusion metal or its alloy and an anode are arranged in an electrolytic bath. An ion exchange membrane is installed between the anode and cathode as a solid electrolyte. An independent claim is included for the manufacturing method of **H2O2** solution which involves supplying ultra-pure water and oxygen content gas to the reaction chamber. ~~An electrolysis is performed.~~ Atomic hydrogen generated by electrolysis is combined with the oxygen from the oxygen content gas to generate highly pure **H2O2**.

USE - Highly pure **H2O2** solution is used in electronic device and in washing process of liquid crystal.

ADVANTAGE - A high purity **hydrogen peroxide** is obtained. The **melting** of anolyte or catholyte is eliminated to use ion exchange membrane as solid electrolyte. The mixing of electrolyte with impurity is prevented. **H2O2** solution is manufactured economically.

Dwg.1/1

FS CPI EPI

FA AB; GI; DCN

MC CPI: E31-E; J03-B; L03-J  
EPI: U11-C06A1B; U14-K01A5

DRN 1732-P; 1740-S; 1779-S

CMC UPB 19990127

M3 \*01\* C101 C408 C550 C730 C800 C801 C802 C804 C805 C807 M411 M424  
M720 M740 M903 M904 M910 N120 N209 N262 N382 N513 Q454  
DCN: R01732-K; R01732-P

L68 ANSWER 8 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1998-014485 [02] WPIDS

X

DNC C1998-005253  
TI Purification of aqueous **hydrogen peroxide**  
solution - by freezing, preparing aqueous solution and passing  
through cation and anion exchange resins.  
DC E36 J01  
PA (MITN) MITSUBISHI GAS CHEM CO INC  
CYC 1  
PI JP 09278417 A 19971028 (199802)\* 5p C01B015-013  
ADT JP 09278417 A JP 1996-92554 19960415  
PRAI JP 1996-92554 19960415  
IC ICM C01B015-013  
ICS B01D009-02  
AB JP 09278417 A UPAB: 19980112

Aqueous soln. (A) contg. at least 45.2 wt.% of **hydrogen peroxide** is cooled to minus 0.5-56.1 deg. C to deposit solid phase (B), from which aqueous soln. contg. 10-70 wt.% of **hydrogen peroxide** is prepared. The aqueous solution is then passed through at least two layers of cation exchange resin (C), anion exchange resin (D), and a mixture of (C) and (D).

The rate of deposition of (B) is 18-150 g per 1 litre of (A) per hour. **Hydrogen peroxide** concn. of (A) is 45.2-61.2 wt.%.

In an example, an aqueous soln. contg. 55 wt.% of **hydrogen peroxide** was cooled to minus 53 deg. C, added with seed **crystals**, and then further cooled to minus 54.5 deg. C, keeping the deposition rate of the solid at 60 g per 1 litre of the soln. per hour. Aqueous soln. contg. 31 wt.% of **hydrogen peroxide** was prepared by melting the solid and then diluting it. The solution was treated with (B), (C), and (D). TOC of the product was 0.6 ppb.

USE - Production of high purity aqueous **hydrogen peroxide** solution.

ADVANTAGE - The method is able to reduce the concn. of impurities in the aqueous soln. of **hydrogen peroxide** to less than 1/10 of that for conventional products.

Dwg.0/0

FS CPI  
FA AB; DCN  
MC CPI: E11-Q01; E31-E; J01-D04  
DRN 1732-P; 1732-S  
CMC UPB 19980216  
M3 \*01\* C101 C408 C550 C730 C800 C801 C802 C804 C805 C807 M411 M720  
M903 M904 M910 N163 N470 N511 Q431 Q507 R023  
DCN: R01732-P

L68 ANSWER 9 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 1994-316094 [39] WPIDS  
CR 1999-094746 [08]; 1999-507704 [39]  
DNC C1994-143961  
TI Bleaching of kraft wood pulp after brown stock washing - by a

multistage process giving reduced environmental impact.

DC F09  
 IN AMBADY, R; MAPLES, G E  
 PA (USPL) CHAMPION INT CORP; (AMBA-I) AMBADY R; (MAPL-I) MAPLES G E  
 CYC 25  
 PI US 5352332 A 19941004 (199439)\* 14p D21C011-04  
 WO 9600322 A1 19960104 (199607)# EN 52p D21C009-147  
 RW: AT BE CH DE DK ES FR GB GR IE IT LU MC NL PT SE  
 W: AU BR CA FI JP KR NZ  
 AU 9473211 A 19960119 (199616)# D21C009-147  
 EP 739434 A1 19961030 (199648)# EN D21C009-147  
 R: AT BE CH DE DK ES FR GB GR IE IT LI LU MC NL PT SE  
 FI 9605207 A 19970221 (199721)# D21C000-00  
 BR 9408596 A 19970819 (199739)# D21C009-147  
 AU 685483 B 19980122 (199811)# D21C009-147  
 EP 831170 A2 19980325 (199816)# EN 17p D21C009-147  
 R: AT BE CH DE DK ES FR GB GR IE IT LI LU MC NL PT SE  
 JP 10502138 W 19980224 (199818)# 37p D21C011-00  
 KR 97704091 A 19970809 (199836)# D21C009-147  
 NZ 269362 A 19980728 (199836)# D21C011-00  
 EP 739434 B1 19990127 (199909)# EN D21C009-147  
 R: AT BE CH DE DK ES FR GB GR IE IT LI LU MC NL PT SE  
 DE 69416329 E 19990311 (199916)# D21C009-147  
 ADT US 5352332 A Cont of US 1991-646179 19910128, CIP of US 1992-873673  
 19920424, US 1992-944327 19920914; WO 9600322 A1 WO 1994-US7450  
 19940627; AU 9473211 A AU 1994-73211 19940627, WO 1994-US7450  
 19940627; EP 739434 A1 EP 1994-923306 19940627, WO 1994-US7450  
 19940627; FI 9605207 A WO 1994-US7450 19940627, FI 1996-5207  
 19961223; BR 9408596 A BR 1994-8596 19940627, WO 1994-US7450  
 19940627; AU 685483 B AU 1994-73211 19940627; EP 831170 A2 Div ex EP  
 1994-923306 19940627, EP 1997-116682 19940627; JP 10502138 W WO  
 1994-US7450 19940627, JP 1996-503092 19940627; KR 97704091 A WO  
 1994-US7450 19940627, KR 1996-707499 19961227; NZ 269362 A NZ  
 1994-269362 19940627, WO 1994-US7450 19940627; EP 739434 B1 EP  
 1994-923306 19940627, WO 1994-US7450 19940627, Related to EP  
 1997-116682 19940627; DE 69416329 E DE 1994-616329 19940627, EP  
 1994-923306 19940627, WO 1994-US7450 19940627  
 FDT AU 9473211 A Based on WO 9600322; EP 739434 A1 Based on WO 9600322;  
 BR 9408596 A Based on WO 9600322; AU 685483 B Previous Publ. AU  
 9473211, Based on WO 9600322; EP 831170 A2 Div ex EP 739434; JP  
 10502138 W Based on WO 9600322; KR 97704091 A Based on WO 9600322;  
 NZ 269362 A Based on WO 9600322; EP 739434 B1 Related to EP 831170,  
 Based on WO 9600322; DE 69416329 E Based on EP 739434, Based on WO  
 9600322  
 PRAI US 1992-944327 19920914; US 1991-646179 19910128; US 1992-873673  
 19920424; WO 1994-US7450 19940627; AU 1994-73211 19940627; EP  
 1994-923306 19940627; FI 1996-5207 19961223; BR 1994-8596  
 19940627; EP 1997-116682 19940627; JP 1996-503092 19940627; KR  
 1996-707499 19961227; NZ 1994-269362 19940627; DE 1994-616329  
 19940627  
 REP US 4269656; US 4619733; US 4946556  
 IC ICM D21C000-00; D21C011-00; D21C011-04



ICS D21C009-02; D21C009-10; D21C009-12; D21C011-10

ICA D21C009-14; D21C009-147

AB US 5352332 A UPAB: 19991014

Kraft wood pulp is bleached after brown stock washing by (i) oxygen delignification in the presence of O<sub>2</sub> and oxidised white liquor; (ii) washing once or twice; (iii) acid treating at pH 2-4 to remove non-process metals; (iv) washing the pulp with fresh water; (v) discharging (some of) the filtrate to waste; (vi) ClO<sub>2</sub> bleaching at pH 2-4 and a consistency of 9-14%; (vii) partially neutralising to pH 5-7 with filtrate from (xi) and with oxidised white liquor; (viii) thickening pulp on a decker; (ix) adding oxidised white liquor to give pH 10-13; (x) adding O<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> bleaching chemicals; (xi) subjecting the thickened pulp and bleaching chemicals to hot extraction; (xii) thoroughly washing pulp with fresh water on two sequential washing stages to minimise the wash volume; (xiii) subjecting the pulp to 1 or 2 final ClO<sub>2</sub> bleaching stages and, after each stage, washing thoroughly with fresh water and/or paper machine white water; (xiv) discharging each washing filtrate to waste; (xv) using filtrate from (xii) to dilute and neutralise pulp from (vi); (xvi) sepg. the combined filtrates from the pulp on the decker; (xvii) recycling (some of) the filtrate to (ii) as wash water; (xviii) recovering filtrate from the last washer of (ii) in the cooking liquor recovery process by recycling **countercurrently** through brown stock washing constituting a weak black liquor, which is (xix) evaporated to a strong black liquor; (xx) burning this liquor in a recovery boiler to produce a smelt and a flue gas, such that chloride from (xv) is preferentially enriched in the flue gas; (xii) collecting particles contg. chloride and sulphate from the flue gas as ash in an electrostatic precipitator; (xxii) removing Cl and K from the ash by (a) leaching with water or (b) evaporation **crystallisation** from water soln. to give a solid Na<sub>2</sub>SO<sub>4</sub> stream and a KCl soln.; (xxiii) discharging the KCl soln. or recovering it for its Cl content; (xxiv) mixing the Na<sub>2</sub>SO<sub>4</sub> with the strong black liquor before (xx); and (xxv) dissolving the recovery boiler smelt in water to form green liquor and converting this to pulping liquor in the causticising process.

ADVANTAGE - Process reduces the environmental impact of bleached wood pulp mfr.

Dwg.2/4

FS CPI

FA AB; GI

MC CPI: F05-A02B

DRN 1287-S; 1514-S; 1678-P; 1732-S; 1744-P; 1779-S; 1896-S

L68 ANSWER 10 OF 10 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1981-22633D [13] WPIDS

TI Mfg. hot melt adhesive - by oxidising polybutadiene with **hydrogen peroxide** or organic peracid.

DC A12 A81 G03

PA (HIRO-N) HIRONO KAGAKU KOGYO

CYC 1

PI JP 56009924 B 19810304 (198113)\*  
JP 54050536 A 19790420 (198113)  
PRAI JP 1977-117662 19770929  
IC C08F008-08; C09J003-14  
AB JP 81009924 B UPAB: 19930915  
Method comprises oxidising polybutadiene having mol. wt. of approx.  
70000, ~~1.2-content of at least 70% and degree of~~  
**crystallinity** of at least 10% with **H2O2** or organic  
peracid obtd. from **H2O2** and organic acid.  
Used for bonding wood, fabric, plaster board, glass ceramic,  
metal, etc. (J54050536)  
FS CPI  
FA AB  
MC CPI: A04-B02; A10-E11; A12-A05A; G03-B02B  
PLC UPA 19930924  
KS: 0229 0231 1093 2010 2198 2203 2583 2585 2642 2684 2723 2728 2729  
FG: \*001\* 011 03- 04- 117 122 231 247 359 36& 440 445 47& 477 575  
577 578 583 585 587 589 609 688

=> d 169 1-6 max

L69 ANSWER 1 OF 6 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 1999-469682 [40] WPIDS  
DNC C1999-138005  
TI Resin composition for extrusion blow moulding - prepared from LLDPE  
resin and hydrogen peroxide.  
DC A17 A32 A97  
IN LI, Y; WANG, D; XU, D  
PA (CHPE-N) CHINA PETROCHEMICAL GEN CO  
CYC 1  
PI CN 1218066 A 19990602 (199940)\* C08L023-06  
ADT CN 1218066 A CN 1997-119782 19971113  
PRAI CN 1997-119782 19971113  
IC ICM C08L023-06  
ICS C08K005-14  
AB CN 1218066 A UPAB: 19991004  
A resin composition for extruding out and blowing the agricultural  
ground film is prepared from LLDPE resin with 0.88-0.95 g/cu.cm of  
density and 0.5-3 g/10min of flow rate for **molten** one and  
**hydrogen peroxide** (500-10000 ppm) through a  
dual-screw extrusion.  
ADVANTAGE - A film of 10 microns in thickness can be blown  
with stable physical nature.  
FS CPI  
FA AB  
MC CPI: A04-G06; A08-C05; A11-B07A; A12-W04A

L69 ANSWER 2 OF 6 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 1999-199175 [17] WPIDS  
DNC C1999-058368

TI Bleaching detergent composition - comprises compound capable of releasing hydrogen peroxide and bleaching activating agent.

DC D25 E19

PA (KAOS) KAO CORP

CYC 1

PI JP 11043691 A 19990216 (199917)\* 10p C11D003-395

ADT JP 11043691 A JP 1997-203567 19970729

PRAI JP 1997-203567 19970729

IC ICM C11D003-395

ICS C11D001-72; C11D003-20; C11D003-34; C11D003-39; C11D017-06

AB JP 11043691 A UPAB: 19990503

Bleaching detergent composition includes: (i) an organic compound (A), stable against **hydrogen peroxide**, having the **melting** point of not more than 70 deg. C and being capable of releasing hydrogen peroxide in water; and (ii) a bleaching activating agent (B) expressed by the following formula (1):

Formula (1)-p

in which R is a 7-15C alkyl group; Y is -SO<sub>3</sub>M or -COOM; and M is H or an alkali metal atom.

USE - The detergent composition is used for washing clothes.

ADVANTAGE - Fine powder **produced** when the **hydrogen-peroxide** releasing ingredient is decomposed is adsorbed onto the nonionic surface active agent. Contact between this fine powder and the bleaching activating agent is prevented to avoid decomposition of the bleaching activating agent.

Dwg.0/0.

FS CPI

FA AB; GI; DCN

MC CPI: D11-A03; D11-B01A; D11-B01B; D11-B01D; E10-A09B7; E10-C03; E33-D

CMC UPB 19990525

M3 \*01\* G011 G012 G013 G100 J0 J011 J012 J131 J2 J241 K431 M220 M221 M222 M223 M224 M225 M226 M231 M232 M233 M262 M281 M320 M414 M510 M520 M531 M540 M630 M781 M903 M904 Q273 Q507 R036

DCN: 9917-FAR01-K; 9917-FAR01-U

L69 ANSWER 3 OF 6 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1991-038096 [06] WPIDS

DNN N1991-029422 DNC C1991-016301

TI Physical and chemical surface treatment - by ice particle blasting and hydrogen peroxide treatment.

DC L03 P43 P61 U11

IN ENDO, S; FUKUMOTO, T; NAMBA, K; OMORI, T

PA (MITQ) MITSUBISHI DENKI KK

CYC 3

PI DE 4022401 A 19910131 (199106)\* 10p

JP 03049224 A 19910304 (199115)

DE 4022401 C 19920102 (199201)

US 5081068 A 19920114 (199206)

ADT DE 4022401 A DE 1990-4022401 19900713; JP 03049224 A JP 1989-185243

19890717; US 5081068 A US 1990-546446 19900703  
PRAI JP 1989-185243 19890717  
IC B08B007-00; B24C001-00; B24C003-32; H01L021-30  
AB DE 4022401 A UPAB: 19970205

A substrate surface treatment process involves one of the following:  
(a) (i) physical treatment by blasting with hydrogen peroxide-contg. ice particles and (ii) chemical treatment with **hydrogen peroxide soln. formed by melting** of the ice particles; (b) (i) physical treatment by blasting with pure ice particles and (ii) chemical treatment by applying a hydrogen peroxide soln. to the surface; or (c) (i) physical treatment by blasting with pure ice particles and (ii) chemical treatment by applying a hydrogen peroxide-contg. gas onto the surface.

USE/ADVANTAGE - The process is useful for surface cleaning, surface prepn., resist removal or etching of substrates used in electronic components, e.g. semiconductor devices. It provides effective surface treatment by a combination of physical and chemical treatment.

Dwg.1/6

ABEQ US 5081068 A UPAB: 19930928

Substrates e.g. for semiconductor devices are surface treated in a container (14) to which H2O2 and water are supplied from an electrode structure (19) supplied with H2 and O2, with N2. The H2O2 water are condensed in a nozzle (6) and become ice particles contg. H2O2. The particles are jetted on to the substrate (12) by a blasting device (11), so contaminants particles and dirt (13) are physically removed.

ADVANTAGE - Chemical cleaning surface reformed etc. can be performed with physical cleaning in the same device.

FS CPI EPI GMPI  
FA AB; GI  
MC CPI: L03-H04E2; L03-H04E9; L04-C09  
EPI: U11-C04A1  
DRN 1532-S; 1732-P; 1779-S

L69 ANSWER 4 OF 6 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1986-021108 [04] WPIDS

DNC C1986-008925

TI Selenium sepn. from urethane solns - by addn. of aromatic hydrocarbon (deriv.) and distn. or fusion, contact with aq. **hydrogen peroxide** soln. and selenium, recovery..

DC E19 E36 L03

PA (ORSI-I) ORSINI J

CYC 1

PI CH 652996 A 19851213 (198604)\* 7p

ADT CH 652996 A CH 1982-3660 19820614

PRAI CH 1982-3660 19820614

IC C01B019-02

AB CH 652996 A UPAB: 19930922

In the recovery of Se from Se-contg. urethane solns., prepd. by the Se-catalysed reaction of an organic cpd. contg. at least one OH gp.

with CO and a nitrated organic cpd. at high temps. and pressure in the presence of a base and/or water, (i) the soln. of urethane reaction prods. contg. Se is distilled between room temp. and 200 deg.C, to remove unreacted hydroxylated cpds., all the aminated bases and/or all water present. (ii) A water-insoluble aromatic hydrocarbon or a nitrated, phenylated, alkylated or halogenated hydrocarbon is added to distilled urethane soln., to form a hydrocarbon-urethane soln. contg. Se. (iii) The hydrocarbon-urethane-Se soln. is contacted, at 25-120 (60-90) deg.C with aq. H2O2 soln. having H2O2 concn. 0.5-50 (3-30) wt.%, to react with the Se cpds. and extract of these cpds. from soln. and **form** an aq.

**H2O2** phase contg. Se and a hydrocarbon phase contg. the urethane. (iv) The Se-contg. aq. H2O2 phase is sepd. from the hydrocarbon phase. (v) The aq. H2O2 phase is treated to separate the Se cpds. and (vi) Se is recovered. Pref. the distilled Se-contg. urethane of step (i) is heated up to 120 deg.C in the complete absence of solvent, to form a melted urethane soln. contg. Se. The melted urethane soln. contg. Se is contacted, between the m.pt. of urethane and 120 deg.C, with an aq. soln. of H2O2, and extract these Se cpds. from the melted urethane soln. to **form** an aq.

**H2O2** phase contg. Se and an organic urethane phase. The Se cpds. are sepd. from the aq. phase and the Se is recovered. USE/ADVANTAGE - The Se can be used in the prodn. of a photo-battery. A large amt. of Se is recovered for re-use e.g. as catalyst. The urethane is purified.

0/0

FS CPI  
FA AB  
MC CPI: E11-Q01; E31-G; L04-A03  
DRN 0679-U; 1732-U; 1780-P  
CMC UPB 19930924  
M3 \*01\* B134 C810 M411 M720 M903 M910 N163 N512 N513 Q421

L69 ANSWER 5 OF 6 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1982-C0477E [08] WPIDS

TI ~~Etching method for printed circuit mfr. - uses hydrogen peroxide and hydrochloric acid according to melting copper volume.~~

DC V04

PA (HOMA-N) HOMA DENSHI KK

CYC 1

PI JP 57010288 A 19820119 (198208)\* 5p

PRAI JP 1980-83778 19800619

IC H05K003-06

FS EPI

FA NOAB

L69 ANSWER 6 OF 6 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1977-73305Y [41] WPIDS

TI Fused acrylonitrile polymers **prepn.** - using **hydrogen peroxide** as initiator to decrease environmental pollution.

DC A14 E36  
PA (JAPE) JAPAN EXLAN CO LTD  
CYC 3  
PI JP 52103487 A 19770830 (197741)\*  
US 4080494 A 19780321 (197817)  
GB 1565273 A 19800416 (198016)  
JP 62000925 B 19870110 (198705)  
PRAI JP 1976-21582 19760227  
IC C08F002-02; C08F004-32; C08F020-44  
AB JP 52103487 A UPAB: 19930901

Fused acrylonitrile polymers are prepd. by polymerising a mixt. of monomers consisting of acrylonitrile or principally acrylonitrile and at least one of any other ethylene type unsatd. cpds., in the presence of 3-80 wt.% of water relative to the total amt. of the monomers and water at  $\geq 80$  degrees C under pressure. Hydrogen peroxide is used as the polymerisation initiator, pref. 0.1-8 wt.% relative to the monomer.

Use of hydrogen peroxide as the initiator gives no pollution to the product polymer, thus no fear of causing environmental pollution such as increasing COD or BOD in the waste water. Moreover, the product is colourless. The reaction is quite stable and processes without difficulty. The polymerisation velocity scarcely depends on the reaction temp.

FS CPI  
FA AB  
MC CPI: A02-A01; A04-D03A; E31-E  
PLC UPA 19930924  
FG: \*001\* 010 02- 034 04& 072 074 076 264 266 267 27& 28& 347 348  
516 518 528 679 688 691  
CMC UPB 19930924  
M3 \*01\* C800 C730 C101 C408 C802 C807 C805 C804 C801 C550 Q121 M781  
R021 R022 R023 R024 M411 M902

=> d l81 1-4 max

L81 ANSWER 1 OF 4 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 1999-394628 [33] WPIDS  
DNC C1999-115908  
TI Reduction of catalase activity in alcohol oxidase.  
DC D16 E14 E17  
IN FISH, W W; LITTLE, T M  
PA (MOLD-N) MOLLDOWAN LAB INC  
CYC 1  
PI US 5919684 A 19990706 (199933)\* 5p C12N009-04  
ADT US 5919684 A US 1991-667166 19910311  
PRAI US 1991-667166 19910311  
IC ICM C12N009-04  
ICS C12N009-08  
AB US 5919684 A UPAB: 19990902  
NOVELTY - Reduction of catalase activity in alcohol oxidase  
composition comprises aging composition at specified temperature to

activate catalase while maintaining alcohol oxidase activity.

DETAILED DESCRIPTION - A process for inactivating catalase in a composition comprising alcohol oxidase and catalase comprises aging the composition obtained by the fermentation of *Pichia pastoris* cells at a temperature to accomplish inactivating the catalase while maintaining alcohol oxidase activity.

USE - Alcohol oxidases are used to remove oxygen from compatible solutions, and in **production** of aldehydes and **hydrogen peroxides**.

ADVANTAGE - By removing catalase activity, the use of alcohol oxidase originally high in the catalase activity is enabled, rather than it being discarded.

Dwg.0/0

TECH US 5919684 A UPTX: 19990819

TECHNOLOGY FOCUS - ENZYME - Preferred Process: The composition is prepared by fermenting *Pichia pastoris* cells, lysing the resulting cells and removing cell debris. The alcohol oxidase is crystallized and **crystalline** precipitate **suspended** in phosphate buffer and dissolved in aqueous medium of pH 6-8.5 containing 30 wt% sucrose. A preservative selected from azide (a salt of hydrazoic acid and an electropositive metal selected from Na, K, Ca, Mg and Li, especially sodium azide) or thymol is added in an amount of 0.01-1 wt/vol% to the composition prior to aging. The aging is carried out at 1-15 (especially 4)degreesC for at least 20 (especially 40) days.

KW [1] 2869-0-0-0 CL; 4540-0-0-0 CL; 2582-0-0-0 CL; 60506-0-0-0 CL; 2853-1-0-0 CL; 107316-0-0-0 CL; 217361-0-0-0 CL

FS CPI

FA AB; DCN

MC CPI: D05-A02A; D05-H; E10-D01C; E11-Q02; E31-D02; E31-E

DRN 0135-U; 0558-U; 2052-U

CMC UPB 19990902

M3	*01*	A220	A940	C107	C307	C520	C730	C801	C802	C803	C804	C806	C807
		M411	M782	M904	M905	N134	Q232	Q620	R023				
		DCN: R20476-K; R20476-M											
M3	*02*	A103	A940	C107	C307	C520	C730	C801	C802	C803	C804	C806	C807
		M411	M782	M904	M905	N134	Q232	Q620	R023				
		DCN: R08101-K; R08101-M											
M3	*03*	A119	A940	C101	C107	C307	C520	C730	C801	C802	C804	C806	C807
		M411	M782	M904	M905	N134	Q232	Q620	R023				
		DCN: R08100-K; R08100-M											
M3	*04*	G015	G100	H4	H401	H441	H8	M210	M211	M213	M232	M240	M282
		M320	M414	M510	M520	M531	M540	M782	M904	M905	M910	N134	Q232
		Q620 R023											
		DCN: R00558-K; R00558-M											
M3	*05*	F012	F013	F014	F015	F016	F017	F019	F113	F123	H4	H405	H424
		H483	H5	H521	H8	K0	L8	L814	L818	L822	L831	M1	M126
		M141	M280	M311	M323	M342	M373	M393	M413	M510	M522	M530	M540
		M782	M904	M905	M910	N134	Q232	Q620	R023				
		DCN: R00135-K; R00135-M											
M3	*06*	A111	A940	C107	C307	C520	C730	C801	C802	C803	C804	C806	C807
		M411	M782	M904	M905	M910	N134	Q232	Q620	R023			

DCN: R02052-K; R02052-M  
M3 \*07\* A212 A940 C101 C107 C307 C520 C730 C801 C802 C804 C806 C807  
M411 M782 M904 M905 N134 Q232 Q620 R023  
DCN: RA0CTI-K; RA0CTI-M

L81 ANSWER 2 OF 4 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 1982-51124E [25] WPIDS

TI Sodium percarbonate **production** - from **hydrogen peroxide**, N,N,N',N'-tetra (phosphonomethyl) -di aminoalkane.

AW PHOSPHONO METHYL.

DC E11 E34

PA (MITN) MITSUBISHI GAS CHEM IND CO LTD

CYC 3

PI JP 57077008 A 19820514 (198225)\* 14p

DE 3142574 A 19820902 (198236)

US 4388287 A 19830614 (198326)

JP 59006802 B 19840214 (198410)

DE 3142574 C 19851010 (198542)

ADT JP 57077008 A JP 1980-151004 19801028; DE 3142574 A DE 1981-3142574  
19811027

PRAI JP 1980-150502 19801027; JP 1980-151004 19801028

IC C01B015-10; C01B031-00; C09K015-32

AB JP 57077008 A UPAB: 19930915

In the prodn. of sodium percarbonate (I) from NaCO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> one or more N,N,N',N'-tetra(phosphonomethyl) -diamino alkanes of formula are added to the reaction mixtures. In (II) R is -CHMe-(CH<sub>2</sub>)<sub>n</sub>-; or -CHMe-(CH<sub>2</sub>)<sub>n</sub>-CHMe- and n is 0 or 1-4.

Pref. (II) is N,N,N',N'-tetra-(phosphono methyl) -1,2-diamino -propane and/or -butane. The amt. of (II) added is 100-1000 ppm per that of the reaction mixt. (II) is prepd. by dropwise pouring of aldehyde into a mixture of diamino-alkane, phosphorus acid and HCl.

(I) is produced inexpensively in a high yield. It is stable and can be stored for a long period of time.

ABEQ DE 3142574 C UPAB: 19930915

Sodium percarbonate is prepd. by reacting sodium carbonate with hydrogen peroxide in a working soln. of sodium carbonate, hydrogen peroxide and water and in the presence of a stabiliser for the hydrogen peroxide which is at least one N,N,N',N'-tetra-(phosphonomethyl)- diaminoalkane of formula ((OH)2P(=O)CH<sub>2</sub>)<sub>2</sub>-N-R-N-(CH<sub>2</sub>P(=O)(OH)<sub>2</sub>)<sub>2</sub> (where R is -C(CH<sub>3</sub>)H-(CH<sub>2</sub>)<sub>n</sub>- or -C(CH<sub>3</sub>)H-(CH<sub>2</sub>)<sub>n</sub>-C(CH<sub>3</sub>)H- and n is 0,1,2,3 or 4). An inorganic phosphate silicate and/or magnesium salt is pref. also present. Sodium percarbonate is obtd. aq. **crystalline suspension** which is recovered and dried.

ADVANTAGE - Decomposition of the hydrogen peroxide is minimal.

FS CPI

FA AB

MC CPI: E05-G03C; E31-E

CMC UPB 19930924

M3 \*01\* A111 A940 C106 C408 C530 C730 C801 C802 C803 C805 C807 M411  
M720 M782 M903 N105 N341 N382 R032

M3 \*02\* B415 B702 B713 B720 B744 B815 B833 H1 H103 H182 M280 M311



M312 M313 M314 M315 M321 M323 M331 M333 M340 M342 M361 M383  
M391 M393 M411 M510 M520 M530 M540 M620 M781 M903 Q620

L81 ANSWER 3 OF 4 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 1979-20109B [11] WPIDS  
TI Continuous thiourea di oxide prodn. from thiourea - by oxidn. with  
hydrogen peroxide in aq. medium.

DC A60 B05 E16  
PA (SUDD) SKW TROSTBERG AG  
CYC 1

PI DE 2736943 A 19790308 (197911)\*  
DE 2736943 C 19860507 (198619)

PRAI DE 1977-2736943 19770817

IC C07C157-02; C07C161-00; C09K015-28

AB DE 2736943 A UPAB: 19930901

Prepn. of thiourea dioxide of formula (I) comprises reacting  
thiourea (II) and H<sub>2</sub>O<sub>2</sub> in aq. soln. in an externally cooled  
internally stirred reactant.

Solid (II) and commercial concn. (pref. 30-70 wt%) H<sub>2</sub>O<sub>2</sub> are  
continuously supplied to a reactor filled with reaction soln. Mole  
ratio (II): H<sub>2</sub>O<sub>2</sub> is 1-1.5:1. The obtd. (I) is withdrawn from the  
reactor as a **crystal suspension** and recovered by  
conventional methods.

(I) is a strong reducing agent in acid or esp. ammoniacal soln.  
it can be used e.g. for reducing vat dyes and ketones, as  
polymerisation catalyst for unsatd. Cpds. as a antioxidant and as a  
starting material and auxiliary in the prodn. of pharmaceuticals and  
chemicals.

(I) can be prep'd. continuously in >90% yield and approx. 98%  
purity in a reactor of very low specific vol.

FS CPI  
FA AB  
MC CPI: A02-A; B10-A09C; E10-A09C; E10-A10  
PLC UPA 19930924

KS: 0034 0206 0215 0229 2043 2066 2067 2199 2203 2239 2262 2266

FG: \*001\* 011 03- 247 260 273 293 329 352 360 546 689 691  
CMC UPB 19930924

RIN 00034 00206 00215 00229 02043 02066 02067 02199 02203 02239  
02262 02266

M2 \*01\* K0 M320 M280 C216 K433 L260 M620 N050 M510 M520 M530 M540  
M720 M416 M902

M2 \*02\* K0 M320 M280 C316 K442 L420 M620 N050 M510 M520 M530 M540  
M720 M416 M902

M3 \*03\* K0 M320 M280 C216 K433 L260 M620 N050 Q121 M510 M520 M530  
M540 M720 Q624 Q509 M416 M902

M3 \*04\* K0 M320 M280 C316 K442 L420 M620 N050 Q121 M510 M520 M530  
M540 M720 Q624 Q509 M416 M902

L81 ANSWER 4 OF 4 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 1977-07454Y [05] WPIDS

TI Sodium ~~peroxycarbonate crystals continuous~~ **prepn.** -- from **hydrogen peroxide** and sodium carbonate soln..

DC D25 E34

PA (FMCC) FMC CORP

CYC 8

PI BE 844167 A 19770117 (197705)\*

DE 2631917 A 19770203 (197706)

NL 7607869 A 19770118 (197706)

JP 52020999 A 19770217 (197713)

SE 7608054 A 19770314 (197713)

FR 2318112 A 19770318 (197717)

CA 1070084 A 19800122 (198006)

IT 1062528 B 19841020 (198506)

PRAI US 1975-596187 19750715

IC B01D009-02; B01J000-00; C01B015-10; C01D007-24; C11D003-39

AB BE 844167 A UPAB: 19930901

Prepn. of stable sodium peroxycarbonate of high apparent specific weight and uniform particle size, comprises continuous crystallisation by the following method:-

(a) 50-90% Aq ~~H2O2~~ and purified Na2CO3 soln. are introduced continuously into a crystallisation zone;

(b) the two are reacted maintaining the concn. of the sodium peroxycarbonate above the saturation level under 0.5-15 hrs;

(c) water is evaporated at 1.5-15 mols/mol of sodium peroxycarbonate produced;

(d) a **crystal suspension** is withdrawn, the crystals sepd. and dried to recover sodium peroxycarbonate of specific weight 0.9-1 g/cm3;

(ee the sepd. mother liquor is treated to decompose H2O2; fresh sodium carbonate is added to form a 25-33% soln. at 27 degrees C or above and

(f) the Na2CO3 soln. is purified to remove Fe and other transition metals, then the treated soln. is passed to stage (a).

The prod. is used as a bleaching agent in detergent compsns. The process gives crystals of high specific weight, which are easily dried and are less friable and have less tendency to segregate in compsns.

FS CPI

FA AB

MC CPI: D11-B01; E31-E

CMC UPB 19930924

M3 \*01\* A111 A940 C730 C101 C108 C106 C408 C803 C802 C807 C805 C801  
C530 C550 N010 N000 Q273 M720 Q507 M411 M902

=> d 182 1-8 max

L82 ANSWER 1 OF 8 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 2002-195945 [25] WPIDS

DNC C2002-060616

TI Preparation of modafinil useful in compositions comprises oxidizing 2-((diphenylmethyl)thio)acetamide with **hydrogen**

+

peroxide, precipitation and separation.

DC B05  
IN ARONHIME, J; CEASU, A; GERSHON, N; LIEBERMAN, A; SINGER, C  
PA (ARON-I) ARONHIME J; (CEAU-I) CEASU A; (GERS-I) GERSHON N; (LIEB-I)  
LIEBERMAN A; (SING-I) SINGER C; (TEVA-N) TEVA PHARM IND LTD;  
(TEVA-N) TEVA PHARM USA INC  
CYC 97  
PI WO 2002010125 A1 20020207 (200225)\* EN 35p C07C317-44  
RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC  
MW MZ NL OA PT SD SE SL SZ TR TZ UG ZW  
W: AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CO CR CU CZ  
DE DK DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP  
KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ  
NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US  
UZ VN YU ZA ZW  
US 2002043207 A1 20020418 (200228) C30B007-00  
AU 2001083008 A 20020213 (200238) C07C317-44  
EP 1309547 A1 20030514 (200333) EN C07C317-44  
R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK  
NL PT RO SE SI TR  
KR 2003029636 A 20030414 (200353) C07C317-44  
ADT WO 2002010125 A1 WO 2001-US23689 20010727; US 2002043207 A1  
Provisional US 2000-221110P 20000727, Provisional US 2000-226491P  
20000818, Provisional US 2000-229160P 20000830, Provisional US  
2000-230088P 20000905, Provisional US 2001-259332P 20010102, US  
2001-916885 20010727; AU 2001083008 A AU 2001-83008 20010727; EP  
1309547 A1 EP 2001-961766 20010727, WO 2001-US23689 20010727; KR  
2003029636 A KR 2003-701061 20030124  
FDT AU 2001083008 A Based on WO 200210125; EP 1309547 A1 Based on WO  
200210125  
PRAI US 2001-259332P 20010102; US 2000-221110P 20000727; US 2000-226491P  
20000818; US 2000-229160P 20000830; US 2000-230088P 20000905; US  
2001-916885 20010727  
IC ICM C07C317-44; C30B007-00  
ICS A61K031-165; C30B021-02; C30B028-06  
AB WO 200210125 A UPAB: 20020418  
NOVELTY - Preparation of modafinil comprises: (a) oxidizing  
2-((diphenylmethyl)thio)acetamide with H<sub>2</sub>O<sub>2</sub> in a mixture  
of mineral acid and alcohol or phase transfer catalyst; (b)  
precipitating a solid containing modafinil from the mixture; and (c)  
separating the mixture from the solid.  
DETAILED DESCRIPTION - INDEPENDENT CLAIMS are included for:  
(A) modafinil containing less than 0.02% of  
2-((diphenylmethyl)sulfonyl)acetamide, 2-  
((diphenylmethyl)sulfinyl)acetic acid or 2-  
((diphenylmethyl)sulfinyl)acetate;  
(B) preparation of modafinil Form I involving: dissolving  
modafinil in a liquid; crystallizing modafinil from the liquid; and  
separating the liquid to obtain modafinil Form I. The liquid is  
acetone, acetonitrile, benzyl alcohol, dimethyl formamide, methanol,  
methyl ethyl ketone and/or pyrrolidone (preferably methanol or  
acetone);

(C) preparation of modafinil Form I by suspending modafinil in ethyl acetate to convert into modafinil Form I, and separating the ethyl acetate;

(D) preparation of modafinil Form I involving: **suspending crystalline** Form II modafinil in a liquid to convert into modafinil Form I, and separating the liquid. The liquid is methyl tert-butyl ether, water and/or isobutyl acetate;

(E) preparation of modafinil Form I by heating Form V or Form VI modafinil to at least 80 deg. C;

(F) crystalline form of modafinil (C1) producing a powder X-ray diffraction pattern with reflections (2 theta ) at 14.3, 17.5, 20.5 and 21.3 plus or minus 0.2;

(G) preparation of (C1) involving: either suspending Form III modafinil in water to convert into (C1) and separating the water; or dissolving modafinil in a liquid selected from ethanol, isopropanol, n-butanol, tert-butanol, methyl isobutyl ketone, ethylene glycol, dioxolane and/or dioxane, crystallizing modafinil from the liquid and separating the liquid;

(H) crystalline form of modafinil (C2) producing a powder X-ray diffraction pattern with reflections (2 theta ) at 7.4, 10.5, 20.0 and 20.5 plus or minus 0.2;

(I) preparation of (C2) involving: dissolving modafinil in a liquid selected from toluene and a mixture of ethanol and dimethylcarbonate, crystallizing modafinil from the liquid and separating the liquid;

(J) crystalline form of modafinil (C3) producing a powder X-ray diffraction pattern with reflections (2 theta ) at 6.9, 10.4, 17.2, 20.3 and 22.7 plus or minus 0.2;

(K) preparation of (C3) involving: dissolving modafinil in a liquid selected from tetrahydrofuran or dimethyl sulfoxide, crystallizing modafinil from the liquid and separating the liquid;

(L) a crystalline hemisolvate of modafinil and dimethylcarbonate;

(M) preparation of the hemisolvate involving: dissolving modafinil in a liquid selected from methylcarbonate, ethanol and dimethylcarbonate, water and dimethylcarbonate or acetone and dimethylcarbonate mixtures; crystallizing modafinil from the liquid and separating the liquid;

(N) crystalline form of modafinil (C4) producing a powder X-ray diffraction pattern with reflections (2 theta ) at 9.3, 18.2 and 20.5 plus or minus 0.2; and

(O) preparation of (C4) involving: suspending Form V modafinil in a liquid selected from water and/or ethanol to convert Form V modafinil into (C4); and separating the liquid.

USE - For preparing modafinil useful in pharmaceutical composition (claimed).

ADVANTAGE - The modafinil is free of 2-((diphenylmethyl)sulfonyl)acetamide. The purity of modafinil in the precipitated solid is at least 99% (preferably 99.5%). The crystalline forms enlarge the repertoire of materials that a formulation scientist has available for designing a pharmaceutical

dosage form. The process yields modafinil free of sulphone products within the limits of UV detection after two crystallizations.

Dwg.0/6

TECH WO 200210125 A1UPTX: 20020418

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Process: Step (a) involves suspending 1 equivalent of 2-((diphenylmethyl)thio)acetamide in an inert liquid organic medium (0.07 - 0.13 g/milliliter), adding mineral acid (0.05 - 0.2 molar equivalents), adding alcohol (2 - 4 equivalents) and H<sub>2</sub>O<sub>2</sub> (1.5 - 4 molar equivalents).

Step (a) additionally involves heating the inert liquid organic medium. The process additionally involves isolating modafinil of at least 99.5% purity from the precipitated solid by single crystallization, and further to at least 99.9% purity by single recrystallization. The purity of modafinil is measured by relative area of peaks in ultraviolet chromatogram detected at 225 nm. The H<sub>2</sub>O<sub>2</sub> is added as a 10 - 50 wt.% solution in water.

Preferred Compounds: The mineral acid is sulfuric acid, perchloric acid or phosphoric acid. The alcohol is isopropanol, tert-butanol or 2-methyl-1-butanol. The mixture additionally includes an inert liquid organic medium selected from methanol, ethanol, ethylene glycol, acetone and/or dimethylcarbonate.

(C1) is denominated as modafinil Form II, and produces a powder X-ray diffraction pattern with reflections (2theta) at 9.1, 10.3, 11.1, 11.9, 14.3, 15.2, 16.4, 17.5, 18.4, 20.5, 21.3, 24.6 and 26.6 +/-0.2.

(C2) is denominated as modafinil Form III, and produces a powder X-ray diffraction pattern with reflections (2theta) at 7.4, 9.0, 10.5, 12.3, 14.2, 14.7, 15.1, 16.4, 18.3, 20.0, 20.5, 21.1, 22.1 and 24.5 +/-0.2.

(C3) is denominated as modafinil Form IV, and produces a powder X-ray diffraction pattern with reflections (2theta) at 6.9, 10.4, 14.1, 17.2, 18.5, 20.3, 20.8, 21.6, 22.7, 25.0, 26.5, 27.6 and 28.5 +/-0.2. The hemisolvate is denominated as modafinil Form V, and produces a powder X-ray diffraction pattern with reflections (2theta) at 7.4, 9.3, 10.5, 12.4, 14.7, 16.2, 18.2, 19.9, 21.5, 22.0, 23.6, 24.5, 25.2, 28.4, 29.5 and 31.8 +/-0.2.

(C4) is denominated as modafinil Form VI, and produces a powder X-ray diffraction pattern with reflections (2theta) at 9.0, 9.3, 10.2, 12.4, 14.2, 14.5, 15.3, 17.5, 18.1, 20.0, 20.5, 21.5, 22.0, 23.5, 24.5 and 25.0 +/-0.2.

KW [1] 101167-0-0-0 CL NEW PRD; 209-0-0-0 CL USE; 7-0-0-0 CL USE; 264-0-0-0 CL USE; 63-0-0-0 CL USE

FS CPI

FA AB; DCN

L82 ANSWER 2 OF 8 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1993-394009 [49] WPIDS

DNC C1993-175700

TI Calcium peroxide prodn. - treats aq. suspension of calcium hydroxide with hydrogen peroxide at specified temp. at specified molar ratio.

X

DC E32 L02 M25 M26  
IN AFANASENKO, L D; BONDAR, V M; SAVOSKINA, A I  
PA (CHRE-R) CHEM REAGENTS & PURE MATERIALS INST  
CYC 1  
PI SU 1778068 A1 19921130 (199349)\* 3p C01B015-043  
ADT SU 1778068 A1 SU 1990-4838667 19900612  
PRAI SU 1990-4838667 19900612  
IC ICM C01B015-043  
AB SU 1778068 A UPAB: 19940126  
In this method an aq. suspension of calcium hydroxide is used and treatment at 40-46 deg.C for 90-150 min. is applied. The molar ratio of hydrogen peroxide to calcium hydroxide is 2.0-2.5. The obtd. hydrated **crystalline** calcium peroxide **suspension** is held at 54-60 deg.C for 40-60 min.  
USE/ADVANTAGE - May be used to obtain calcium peroxide used in extra-furnace alumino-thermal processes and also in the extra furnace and furnace metallothermal processes during the prodn. of refractory and rare metals and alloy compsns. based upon them. The aim is to increase the dispersivity of the finished prod.  
Bul.44/30.11.92  
Dwg.0/0  
FS CPI  
FA AB; DCN  
MC CPI: E31-E; L02-A02; L02-G11; M25-X  
DRN 1502-S; 1732-S  
CMC UPB 19940329  
M3 \*01\* A220 A940 C408 C550 C730 C801 C802 C803 C804 C805 C807 M411  
M720 M903 M904 N513 Q451 Q469 R032  
DCN: R04118-P

L82 ANSWER 3 OF 8 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
AN 1988-217524 [31] WPIDS  
DNC C1988-097295  
TI Refining meta-bromo-benzoic acid - by **suspending**  
**crystals** in water, adjusting to PH 3.5-6 by adding sodium  
hydroxide and filtering crystals.

DC C03 E14  
PA (MITU) MITSUBISHI CHEM IND LTD  
CYC 1  
PI JP 63154642 A 19880627 (198831)\* 3p  
JP 07116099 B2 19951213 (199603) 3p C07C063-70  
ADT JP 63154642 A JP 1986-299564 19861216; JP 07116099 B2 JP 1986-299564 19861216  
FDT JP 07116099 B2 Based on JP 63154642  
PRAI JP 1986-299564 19861216  
IC C07C051-48; C07C063-70  
ICM C07C063-70  
ICS C07C051-48  
AB JP 63154642 A UPAB: 19930923  
Crystal of crude methabromobenzoic acid is obtd. by brominating benzoic acid. The **crystal** is **suspended** in an amt. of 1-20 wt. times the amt. crystal. The pH is adjusted with NaOH

or KOH to 3.5-6 under a temp. of 20-100 deg.C and is then maintained at 3.5-6 for 0.5-2 hours. The crystal is filtered to produce high purity methabromobenzoic acid.

USE/ADVANTAGE - Useful as an intermediate for agricultural chemicals or dyes. Impurities are dissolved and the methabromobenzoic acid is filtered out. The method easily collects methabromobenzoic acid having high purity with high yield.

In an example, 1539g 98% sulphuric acid, 450g benzoic acid, 22.8g iodine and 295.2g bromine are fed into a flask having a reflux condensating tube and a stirrer. The temp. is increased to 40 deg. C to dissolve the mixt. 69.0g 35% **hydrogen peroxide** water, is dropped for 3 hours. The resulting reaction soln. is fed into 12.9kg water and is filtered to produce 305.4g crude methabromobenzoic acid. The methabromobenzoic acid is fed into a beaker. Water is added to the methabromobenzoic acid. The temp. is adjusted to 50-60 deg.C A 25% NaOH aq. soln. is fed into the beaker. The soln. is filtered to generate a crystal. The crystal is dried to produce the crystal of refined methabromobenzoic acid.

0/0

FS CPI

FA AB; DCN

MC CPI: C10-C04C; C11-B; C12-M11H; E10-C04C; E11-Q01

CMC UPB 19930924

M2 \*01\* G012 G100 H6 H603 H641 J0 J011 J1 J131 M280 M320 M414  
M510 M520 M531 M540 M720 M903 M904 N164 N425 N512 N513 Q316  
DCN: R11825-P  
M3 \*01\* G012 G100 H6 H603 H641 J0 J011 J1 J131 M280 M320 M414  
M510 M520 M531 M540 M720 M903 M904 N164 N425 N512 N513 Q316  
DCN: R11825-P

L82 ANSWER 4 OF 8 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1986-326859 [50] WPIDS

DNC C1986-141474

TI Titanium-silicon catalyst - comprising microspheres of oligomeric silica and titanium/silicalite.

DC E19 J04 L02

IN BELUSSI, G; BUONOMO, F; CLERICI, M; ESPOSITO, A; NOTARI, B; ROMANO, U

PA (ENIE) ENICHEM SINTESI SPA

CYC 13

PI EP 200260 A 19861210 (198650)\* EN 11p

R: AT BE CH DE FR GB LI LU NL SE

NO 8601578 A 19861117 (198701)

DK 8601849 A 19861024 (198709)

US 4701428 A 19871020 (198744)

US 4859785 A 19890822 (198942)

EP 200260 B 19900523 (199021)

R: AT BE CH DE FR GB LI LU NL SE

DE 3671389 G 19900628 (199027)

US 4954653 A 19900904 (199038)

ADT EP 200260 A EP 1986-200663 19860418; US 4701428 A US 1986-854890  
19860423; US 4859785 A US 1987-75688 19870720; US 4954653 A US

1989-362509 19890607

PRAI IT 1985-20457 19850423

REP A3...8833; DE 2102597; EP 100119; FR 2471950; GB 2024790; No-SR.Pub  
IC B01J021-06; B01J029-04; B01J035-08; C07C037-00; C07C039-00;  
C07C103-38; C07D301-12

AB EP 200260 A UPAB: 19930922

A Ti-Si catalyst is pref. 5-1000 micron microspheres of oligomeric silica (I) and titanium-silicalite (II) in the molar ratio 0.05-0.11 in which (II) is bonded in a 3-dimensional network with Si-O-Si bridges.

Prepn. of the catalyst is claimed in which crystals of (II) are dispersed at room temp. to 200 (pref. 40-100) deg.C for 0.2-10 hrs. in an aq. soln. of silica and tetra-(1-5C) alkylammonium hydroxide (pref. tetra-propylammonium hydroxide (III)), which is prepd. by hydrolysing a tetra-alkylorthosilicate (pref. tetra-ethylorthosilicate (IV)) in an aq. soln. of the (III), producing a **suspension of crystals** of (II) and (I) which is fast-dried.

USE/ADVANTAGE - The catalyst is used for the introduction of hydroxy gps. into aromatic substrate by H<sub>2</sub>O<sub>2</sub> and in the epoxidation of olefins using H<sub>2</sub>O<sub>2</sub>. The agglomeration of the catalyst increases its activity and selectivity and allows easier recovery from the reaction mixt.

0/6

ABEQ EP 200260 B UPAB: 19930922

Catalyst on the basis of silicon and titanium, characterised in that it is formed by microspheres and is constituted by oligomeric silica and crystals of titanium-silicalite with an oligomeric silica titanium-silicate molar ratio comprised within the range of from 0.05 to 0.11, wherein the crystals of titanium-silicalite are engaged with each other by means of Si-O-Si bridges.

ABEQ US 4701428 A UPAB: 19930922

Catalyst for use in treatment of aromatic hydrocarbons is formed by microspheres, 5-1000 microns dia., composed of oligomeric silica and crystals of Ti silicalite, with a molar ratio of silica:silicalite of 0.05-0.11, such that crystals of the Ti silicalite are encased by Si-O-Si bridges. The catalyst is formed by preparing a soln. of silica and tetraalkyl ammonium hydroxide by hydrolysing in the liq. phase a tetraalkyl orthosilicate in an aq. soln. of tetraalkyl ammonium hydroxide at a temp. in the range room temp. - 200 deg.C for 0.2-10 h. Crystals of Ti silicalite are dispersed in the soln. to form a suspension of oligomeric silica and Ti silicalite, which is rapidly dried.

ADVANTAGE - Catalyst is active and selective and can be recovered without significant loss.

ABEQ US 4859785 A UPAB: 19930922

Zeolite catalyst comprises microspheres (dia. 0.005-1 mm) of oligomeric silica and Ti silicate crystals (molar ratio 0.05-0.11), in which the Ti silicate crystals are engaged through Si-O-Si bridges.

USE - The prods. are selective catalysts for hydroxylation of aromatics, (e.g. hydrocarbons, phenols, PhCl, PhNO<sub>2</sub> and PhNHAc)



and/or for the reaction of alkenes with H<sub>2</sub>O<sub>2</sub> in an inert solvent at 80-120deg.C to form alkene oxides.

ABEQ US 4954653 A UPAB: 19930922

Hydroxylation of aromatic hydrocarbons comprises reacting a hydrocarbon with **hydrogen peroxide** in presence of a catalyst comprising microspheres consisting of oligomeric silica and crystals of Ti-silicalite with an oligomeric silica/Ti-silicalite molar ratio in range of 0.05-0.11 in which crystals of Ti silicalite are engaged with each other by means of Si-o-Si bridges.

ADVANTAGE - The small crystals produced can be oxyglometal giving a catalyst which is more active and more selective than single original crystals.

FS CPI

FA AB

MC CPI: E07-A03A; E10-E02; E31-P03; E31-P05A; J04-E04; L02-G; N01-D; N03-B

DRN 0370-P; 0868-S; 0964-S; 1694-S; 1732-S

CMC UPB 19930924

M3 \*01\* B114 B702 B720 B831 C108 C800 C802 C803 C804 C805 C807 M411  
M720 M730 M903 M910 N201 N209 N282 N513 Q421

M3 \*02\* A422 A940 B114 B713 B720 B831 C108 C802 C803 C804 C805 C807  
M411 M720 M730 M903 N201 N209 N282 N513 Q421

M3 \*03\* G010 G011 G012 G013 G100 H341 H4 H401 H402 H441 H442 H541  
H602 H641 H8 J011 J341 M210 M211 M240 M262 M272 M280 M281  
M320 M414 M510 M520 M531 M540 M720 M903 N203 N221 N342 N441  
N513

M3 \*04\* F012 F013 F100 M210 M211 M212 M213 M214 M215 M216 M220 M221  
M222 M223 M224 M225 M226 M231 M232 M233 M240 M280 M281 M282  
M320 M413 M510 M521 M530 M540 M720 M903 N114 N213 N305 N309  
N342 N441 N513

RIN: 00012

L82 ANSWER 5 OF 8 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1983-794083 [42] WPIDS

DNC C1983-102042

TI ~~Prodn. of cadmium sulphate crystals - includes treating aq. soln. with **hydrogen peroxide** and activated carbon and vacuum evapn., used as reagent etc.~~

DC E32

IN BOMSHTTEIN, V E; ZHEREBOVIC, A S

PA (SOLY-I) SOLYUS A S

CYC 1

PI SU 983057 A 19821225 (198342)\* 2p

PRAI SU 1981-3325803 19810731

IC C01G011-00

AB SU 983057 A UPAB: 19930925

Mfr. of ~~crystalline~~ CdSO<sub>4</sub> includes purificn. of the starting soln., evapn. in vacuo and sepn. of the obt'd. suspension. The process is simplified (by omitting the hydration stage) and yields uniform prod. of the desired compsn., by evaporating the soln. at up to 70 deg.C and 10-200 torr.

Typically CdSO<sub>4</sub> soln. (24 kg) of 32 wt.% concn. is purified by the addn. 5 ml H<sub>2</sub>O<sub>2</sub> and 10g activated C, then filtered and evaporated at 22 deg.C under 10 torr vacuum to the start of **crystallisation**. The **suspension** is cooled to room temp. and sepd. by centrifuging, yielding 3.5 kg CdSO<sub>4</sub> crystals, 10 kg mother-liq., and 10.4 kg condensate. The formula of obtd. crystals is 3CdSO<sub>4</sub>.8H<sub>2</sub>O. Bul.47/23.12.82

0/0

FS CPI

FA AB

MC CPI: E35-D

DRN 1669-S; 1732-S; 1904-P

CMC UPB 19930924

M3 \*01\* A548 A940 C101 C108 C316 C540 C730 C801 C802 C803 C804 C805  
M411 M720 M903 M910 N163 N512 N513 N520

L82 ANSWER 6 OF 8 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN

AN 1979-28174B [15] WPIDS

TI Continuous prodn. of crystals from solns. - esp. copper sulphate  
crystals in regenerating copper etching soln., or sodium carbonate  
crystals from cyanide plating soln. (NL 30.3.79).

DC E32 J01 M14

IN STEWARD, F A

PA (REXA) DART IND INC

CYC 8

PI DE 2842303 A 19790405 (197915)\*

GB 2004766 A 19790411 (197915)

NL 7809764 A 19790330 (197916)

JP 54057470 A 19790509 (197925)

FR 2404453 A 19790601 (197927)

US 4159194 A 19790626 (197928)

CA 1083472 A 19800812 (198035)

GB 2004766 B 19820415 (198215)

JP 57055442 B 19821124 (198250)

IT 1099188 B 19850918 (198701)

PRAI US 1977-837134 19770928

IC B01D009-02; B01J000-00

AB DE 2842303 A UPAB: 19930901

The temp. of a soln. (a) is altered by indirect heat exchange in a vessel (1) with a polished interior, to produce a crystal concn. of 5-50, esp. 7-25 wt % a stirrer being used to obtain a suspension (B) of the crystals. The larger crystals settle, and are periodically removed, whereas suspension (b) is fed into a second vessel (2) using gentle stirring so more crystals settle and are pumped into vessel (2) esp. as a suspension contg. 20-70% crystals.

The process is pref. used for either (i) removal of sodium carbonate crystals from spent cyanide plating solns. or (ii) removing CuSO<sub>4</sub>.5H<sub>2</sub>O from a spent Cu etching soln. contg. H<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>O<sub>2</sub> and surplus Cu in water, for regeneration of the soln. The pref. plant includes two ranks fitted with stirrers and drain valves for removing **crystal suspensions**.

FS CPI

FA AB  
 MC CPI: E11-Q; E33-D; E35-A; J01-B; M11-B06; M14-A  
 CMC UPB 19930924  
 M3 \*01\* A111 A940 C730 C108 C106 C316 C803 C802 C807 C805 C804 C801  
 C530 C540 A429 N160 N100 Q431 Q465 M720 R032 R035 R036 M411  
 M902

L82 ANSWER 7 OF 8 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
 AN 1977-64981Y [37] WPIDS  
 TI Regular rhombohedric ~~crystalline sodium percarbonate~~ - obtd. by  
 addn. of conc. ~~hydrogen peroxide to decahydrate~~  
 suspension with polycarboxylic polymer present.

DC A97 E34  
 PA (CAAL) AIR LIQUIDE CANADA LTEE  
 CYC 10  
 PI BE 852255 A 19770909 (197737)\*  
 NL 7703413 A 19771003 (197742)  
 SE 7703515 A 19771024 (197745)  
 JP 52117897 A 19771003 (197746)  
 FR 2355774 A 19780224 (197814)  
 GB 1519431 A 19780726 (197830)  
 US 4118465 A 19781003 (197841)  
 DE 2723563 A 19781130 (197849)  
 IL 51710 A 19810520 (198128)  
 IT 1075575 B 19850422 (198544)  
 JP 61016725 B 19860501 (198622)

PRAI FR 1976-8989 19760329

IC C01B015-10; C01D007-00

AB BE 852255 A UPAB: 19930901

Prepn. of  $\text{Na}_2\text{CO}_3 \cdot 1.5\text{H}_2\text{O}$  in the form of regular rhombohedric crystals comprises reacting conc.  $\text{H}_2\text{O}_2$  with a fluid suspension of  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  in a satd. soln. of  $\text{Na}_2\text{CO}_3$  Decahydrate is in the form of at least partly, regular rhombohedric crystals and is used in an amt. such that the heat of dissolution balances the heat of crystallisation or the formation of the percarbonate and the other phenomena consume the heat prod., in the presence of a crystallisation modifying polycarboxylate polymer.

Pref. 0.2-0.8 wt. % of sulphate ions are present during the decahydrate **crystallisation**. The **suspension** may be prepd. by dissolution of anhydrous solid  $\text{Na}_2\text{CO}_3$  in the mother-liquor form a percarbonate crystallisation at  $\geq 37-38$  degrees C, cooling to  $\leq 32$  degrees C lowering to a temp. such the 15-40 % of solid phase is present.

The mother liq. pref. contains 1.5-2% NaCl. Suitable crystallisation modifiers are copolymers of maleic anhydride or maleic or fumaric acids with ethylene, propylene isobutylene, styrene, the vinyl ether or furan pref. as 0.01-1 g. per mole of  $\text{Na}_2\text{CO}_3$ . esp. 0.03-0.1 g.

Process avoids the tendency for the percarbonate to adhere to cold surfaces and the need to add stabilisers. The process gives high yields due to efficient heat-exchanging. The prod. is easily cast.

FS CPI  
 FA AB  
 MC CPI: A12-W11; E31-E  
 PLC UPA 19930924  
 FG: \*001\* 010 034 04- 041 046 047 075 104 105 106 107 155 157 27&  
 398 57- 623 624 721  
 FG: \*002\* 010 034 04- 041 046 050 075 104 105 106 107 155 157 27&  
 398 57- 623 624 721  
 FG: \*003\* 010 034 04- 041 046 052 075 104 105 106 107 155 157 27&  
 398 57- 623 624 721  
 FG: \*004\* 010 034 04- 055 056 075 104 105 106 107 155 157 27& 398  
 57- 623 624 721  
 FG: \*005\* 010 034 04- 075 091 093 104 105 106 107 155 157 27& 398  
 57- 623 624 721  
 FG: \*006\* 010 034 04- 075 104 105 106 107 134 155 157 27& 398 57-  
 623 624 721 726  
 CMC UPB 19930924  
 M3 \*01\* A111 A940 C730 C101 C108 C408 C803 C802 C807 C805 C801 C530  
 N050 N000 M720 M411 M902  
 L82 ANSWER 8 OF 8 WPIDS COPYRIGHT 2003 THOMSON DERWENT on STN  
 AN 1973-79092U [51] WPIDS  
 TI Sodium perborate tetrahydrate - from metaborate and **hydrogen**  
**peroxide.**  
 DC E34  
 PA (DEGS) DEUT GOLD & SILBER AG  
 CYC 5  
 PI BE 799788 A (197351)\*  
 DE 2243342 A 19740404 (197415)  
 FR 2197813 A 19740503 (197422)  
 DE 2243342 B 19741107 (197446)  
 US 3985862 A 19761012 (197643)  
 CH 590177 A 19770729 (197734)  
 PRAI DE 1972-2243341 19720902; DE 1972-2243342 19720902  
 IC C01B015-12; C01D000-00  
 AB BE 799788 A UPAB: 19930831  
Course attrition-resistant title cpd. (I) is made by reaction of Na  
metaborate (II) with H2O2 in an aq. suspension of seeds of  
(I) in a satd. soln. of (I). A clear supersatd. soln. containing  
200-700 g/l (I) obtd. from (II) and H2O2, at 40-50 degrees  
C, is added to the rst. of the salt suspension in the  
crystallizer, and the process is then performed in known  
manner.  
 FS CPI  
 FA AB  
 MC CPI: E31-E  
 CMC UPB 19930924  
 M3 \*01\* A111 A940 C108 C408 C803 C802 C807 C805 C804 B720 B805 B809  
 B831 B105 B701 B711 N050 N000 M720 Q507 M411 M902

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L66 ANSWER 1 OF 28 HCA COPYRIGHT 2003 ACS on STN

137:8249 **Crystallization** process for **producing**  
highly concentrated **hydrogen peroxide**.

Nordhoff, Stefan; Creutz, Matthias; Gross, Stefan; Wagner, Rudolf  
(Degussa Ag, Germany). PCT Int. Appl. WO 2002044083 A1 20020606, 18  
pp. DESIGNATED STATES: W: AU, BR, CA, IL, JP, MX, NO, NZ, PL, SG,  
SI, ZA; RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU,  
MC, NL, PT, SE, TR. (English). CODEN: PIXXD2. APPLICATION: WO  
2001-EP12293 20011024. PRIORITY: EP 2000-126387 20001202.

AB The invention relates to a process for the continuous prepn. of very  
highly concd. hydrogen peroxide having a content of greater than 80  
wt.%, esp. over 98 wt.%, by suspension **crystn.** and  
after-treatment of the **H2O2 crystals**. According to the  
invention, the after-treatment takes the form of  
**countercurrent** washing in a hydraulic or mech. washing  
column with a packed **crystal** bed. The invention also  
provides hydrogen peroxide having a concn. of from 99.9 to 100 wt.%  
and a content of TOC, nitrate, phosphate, nickel and tin of in each  
case less than 4 mg/L.

IC ICM C01B015-013

CC 49-8 (Industrial Inorganic Chemicals)

ST **crystn** process concd **hydrogen peroxide**  
**prodn**

IT Oxidizing agents.

(**crystn.** process for **producing** highly concd.  
**hydrogen peroxide**)

IT **Crystallization**

(suspension; **crystn.** process for **producing**  
highly concd. **hydrogen peroxide**)

IT 7722-84-1P, **Hydrogen peroxide**,  
**preparation**

(**crystn.** process for **producing** highly concd.  
**hydrogen peroxide**)

L66 ANSWER 2 OF 28 HCA COPYRIGHT 2003 ACS on STN

134:209576 Sugar beet membrane filtration process. Reisig, Richard C.;  
Mannapperuma, Jatal D.; Donovan, Michael; Jansen, Robert P.;  
Hlavacek, Marc; Walker, Gordon; Williams, John C. (Tate & Lyle,  
Inc., USA; Tate & Lyle Industries, Limited). PCT Int. Appl. WO  
2001014594 A2 20010301, 57 pp. DESIGNATED STATES: W: AE, AG, AL,  
AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE,  
DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS,  
JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK,  
MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ,

TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, BF, BJ, CF, CG, CH, CI, CM, CY, DE, DK, ES, FI, FR, GA, GB, GR, IE, IT, LU, MC, ML, MR, NE, NL, PT, SE, SN, TD, TG. (English). CODEN: PIXXD2. APPLICATION: WO 2000-US22301 20000815. PRIORITY: US 1999-376026 19990819; US 2000-618831 20000718; US 2000-618416 20000718.

AB A process for producing sugar from beets, comprises macerating beets or pieces, mech. sepg. juice from the macerated beets, and membrane filtering the sepd. juice, producing a retentate and a permeate. The mech. extn. of juice is done on a moving porous vacuum filtration belt with **countercurrent** flow of macerated beets and water. The pH of the vacuum extd. juice is adjusted .gtorsim.7 by addn. of sodium hydroxide. This process does not use conventional beet diffusion. No lime or CO2 are required to be contacted with the juice or the permeate in this process.

IC ICM C13D

CC 44-4 (Industrial Carbohydrates)

IT Centrifuges

#### Crystallization

Diffusion

Electrodialysis

Evaporation

Ion exchangers

Membrane filtration

Purification

Reverse osmosis

Sugar beet

Ultrafilters

Ultrafiltration

(process for producing sugar from beets by membrane filtration)

IT 1305-62-0, Calcium hydroxide, uses 1310-73-2, Sodium hydroxide, uses 7446-09-5, Sulfur dioxide, uses 7664-93-9, Sulfuric acid, uses 7722-84-1, **Hydrogen peroxide**, uses (process for **producing** sugar from beets by membrane filtration)

L66 ANSWER 3 OF 28 HCA COPYRIGHT 2003 ACS on STN

132:303828 The essential role of H2O2 in the regulation of intracellular Ca2+ by epidermal growth factor in Rat-2 fibroblasts. Lee, Z.-W.; Kweon, S.-M.; Kim, S.-J.; Kim, J.-H.; Cheong, C.; Park, Y.-M.; Ha, K.-S. (Biomolecule Research Group, Korea Basic Science Institute, Taejeon, S. Korea). Cellular Signalling, 12(2), 91-98 (English) 2000. CODEN: CESIEY. ISSN: 0898-6568. Publisher: Elsevier Science Inc..

AB The authors have investigated a new mechanism by which epidermal growth factor (EGF) increases intracellular Ca2+ ([Ca2+]i) in Rat-2 fibroblasts. EGF induced a transient increase of [Ca2+]i, and sustained Ca2+ increase disappeared in the absence of extracellular Ca2+. However, EGF had no effect on the formation of inositol phosphates. Expression of N17Rac or **scrape**-loading of C3 transferase blocked the elevation of [Ca2+]i by EGF, but not by lysophosphatidic acid (LPA). EGF increased intracellular H2O2, with

a maximal increase at 5 min, which was blocked by catalase, **scrape**-loading of C3 transferase, or expression of N17Rac. H2O2 scavengers, catalase and N-acetyl-L-cysteine, also blocked the Ca<sup>2+</sup> response to EGF, but not to LPA. In the presence of EGTA, preincubation with EGF completely inhibited subsequent Ca<sup>2+</sup> response to extracellular H2O2 and vice versa. Incubation with EGF or phosphatidic acid abolished subsequent elevation of [Ca<sup>2+</sup>]<sub>i</sub> by phosphatidic acid or EGF, resp. Furthermore, preincubation with LPA inhibited the subsequent Ca<sup>2+</sup> response to EGF, but not vice versa. These results suggested that intracellular H2O2 regulated by Rac and RhoA, but not inositol phosphates, was responsible for the EGF-stimulated elevation of [Ca<sup>2+</sup>]<sub>i</sub>. It was also suggested that EGF cross talked with LPA in the regulation of [Ca<sup>2+</sup>]<sub>i</sub> by **producing** intracellular H2O2.

CC 2-10 (Mammalian Hormones)

L66 ANSWER 4 OF 28 HCA COPYRIGHT 2003 ACS on STN  
132:9318 ~~Roles of RhoA and phospholipase A2 in the elevation of intracellular H2O2 by transforming growth factor-.beta. in Swiss 3T3 fibroblasts.~~ Koo, H.-Y.; Shin, I.; Lee, Z.-W.; Lee, S.-H.; Kim, S.-H.; Lee, C.-H.; Kang, H.-S.; Ha, K.-S. (Biomolecule Research Group, Korea Basic Science Institute, Taejon, S. Korea). Cellular Signalling, 11(9), 677-683 (English) 1999. CODEN: CESIEY. ISSN: 0898-6568. Publisher: Elsevier Science Inc..

AB The authors have investigated the mechanisms by which transforming growth factor-.beta. (TGF-.beta.) increased intracellular H2O2 in Swiss 3T3 fibroblasts. Increase of intracellular H2O2 by TGF-.beta. was maximal at 30 min and blocked by catalase from Aspergillus niger. **Scrape**-loading of C3 transferase, which down-regulated RhoA, inhibited the **prodn.** of H2O2 in response to TGF-.beta.. TGF-.beta. stimulated release of arachidonic acid, which was completely inhibited by mepacrine, a phospholipase A2 inhibitor. Mepacrine also blocked the increase of H2O2 by TGF-.beta.. In addn., arachidonic acid increased intracellular H2O2. Furthermore, TGF-.beta. stimulated stress fiber formation, which was blocked by catalase, without membrane ruffling. Catalase also inhibited stimulation of thymidine incorporation by TGF-.beta.. These results suggested that TGF-.beta. increased intracellular H2O2 through RhoA and phospholipase A2, and also suggested that intracellular H2O2 was required for the stimulation of stress fiber formation and DNA synthesis in response to TGF-.beta..

CC 2-10 (Mammalian Hormones)

L66 ANSWER 5 OF 28 HCA COPYRIGHT 2003 ACS on STN  
131:211993 ~~Lysophosphatidic acid increases intracellular H2O2 by phospholipase D and RhoA in rat-2 fibroblasts.~~ Shin, Incheol; Kweon, Soo-Mi; Lee, Zee-Won; Kim, Seung Il; Joe, Cheol O.; Kim, Jae-Hong; Park, Young-Mok; Ha, Kwon-Soo (Biomolecule Research Group, Korea Basic Science Institute, Taejon, 305-333, S. Korea). Molecules and Cells, 9(3), 292-299 (English) 1999. CODEN: MOCEEK. ISSN: 1016-8478. Publisher: Springer-Verlag Singapore Pte. Ltd..

AB We have investigated the possible roles of phospholipase D (PLD) and RhoA in the **prodn.** of intracellular **H2O2** and actin polymn. in response to lysophosphatidic acid (LPA) in Rat-2 fibroblasts. LPA increased intracellular **H2O2**, with a maximal increase at 30 min, which was blocked by the catalase from *Aspergillus niger*. The LPA-stimulated **prodn.** of **H2O2** was inhibited by 1-butanol or PKC-downregulation, but not by 2-butanol. Purified phosphatidic acid (PA) also increased intracellular **H2O2** and the increase was inhibited by the catalase. The role of RhoA was studied by the **scrape**-loading of C3 transferase into the cells. The C3 toxin, which inhibited stress fiber formation stimulated by LPA, blocked the **H2O2 prodn.** in response to LPA or PA, but had no inhibitory effect on the activation of PLD by LPA. Exogenous **H2O2** increased F-actin content by stress fiber formation. In addn., catalase inhibited actin polymn. activated by LPA, PA, or **H2O2**, indicated the role of **H2O2** in actin polymn. These results suggest that LPA increased intracellular **H2O2** by the activation of PLD and RhoA, and that intracellular **H2O2** was required for the LPA-stimulated stress fiber formation.

CC 13-2 (Mammalian Biochemistry)

ST lysophosphatidate phosphatidate hydrogen peroxide phospholipase D RhoA fibroblast; actin polymn stress fiber **formation hydrogen peroxide**

L66 ANSWER 6 OF 28 HCA COPYRIGHT 2003 ACS on STN  
 130:359531 Phase transition and decomposition of 90% **hydrogen peroxide** at high pressures. ~~Cynn, Hyunchae; Yoo, Choong-Shik; Sheffield, Stephen A.~~ (Lawrence Livermore National Laboratory, Livermore, CA, 94550, USA). Journal of Chemical Physics, 110(14), 6836-6843 (English) 1999. CODEN: JCPSA6. ISSN: 0021-9606. Publisher: American Institute of Physics.

AB Phys. and chem. changes of 90% **H2O2** were studied to pressures of 12 GPa by using a diamond-anvil cell, synchrotron x-ray diffraction, and Raman spectroscopy. **H2O2** freezes at 1.5 GPa and ambient temp. into a tetragonal structure (space group P41212, Z = 4, denoted as **H2O2-I**). This is the same transition that was previously reported in this material at 253 K. The unit cell parameters at 6.3 GPa are a 3.759, c 7.397 .ANG., representing 21% compression from that at ambient pressure. **H2O2-I** was found to transform into a high pressure phase **H2O2-II** at 7.5 GPa, and it decompn. into **H2O** and **O** at the onset of melting, which may be incongruent. In contrast to **H2O**, **H2O2** exhibits a relatively simple polymorphism and a pos. initial slope of the melting curve at high pressures.

CC 75-7 (Crystallography and Liquid Crystals)  
 Section cross-reference(s): 65

ST phase transition decompn **hydrogen peroxide** solid pressure

IT Decomposition  
 Phase diagram  
 (of 90% **hydrogen peroxide** at high pressures)



- IT **Crystal structure**  
**Melting**  
Polymorphism (**crystal**)  
(of **hydrogen peroxide** solid high pressure phases)
- IT Structural phase transition  
(phase transition and decompn. of 90% **hydrogen peroxide** at high pressures)
- IT 7722-84-1, **Hydrogen peroxide**, properties  
(phase transition and decompn. of 90% **hydrogen peroxide** at high pressures)

L66 ANSWER 7 OF 28 HCA COPYRIGHT 2003 ACS on STN

129:150836 Production of chlorine dioxide. Dufour, Pascal (Elf Atochem S. A., Fr.). Fr. Demande FR 2757500 A1 19980626, 35 pp. (French). CODEN: FRXXBL. APPLICATION: FR 1996-15872 19961223.

AB ClO<sub>2</sub> is produced by redn. of a chlorate with a chloride in a strong acid in an evapn.-**crystn.** reactor at 25-90.degree. and 20-400 torr. PdCl<sub>2</sub>, MnCl<sub>2</sub>, or their organometallic complexes are used as catalysts. A gaseous flow contg. ClO<sub>2</sub> and a byproduct of a **crystd.** salt of the strong acid are formed. The **crystd.** salt is charged downwards into a washing column and contacted **countercurrently** with an ascending satd. salt soln. (e.g., brine) which is fed into the reactor. Washing is done at -5 to 90.degree. (preferably at 15-70.degree.). Optionally, the reaction is performed in the presence of H<sub>2</sub>O<sub>2</sub> to reduce the Cl<sub>2</sub> byproduct to HCl. ClO<sub>2</sub> is suitable for bleaching of paper, textile fibers, wastewater treatment, water purifn., denitrification of waste gases, and decoloration of greases.

IC ICM C01B011-02

ICS B01J023-44; B01J023-34; B01J031-12

CC 49-2 (Industrial Inorganic Chemicals)

Section cross-reference(s): 40, 43, 59, 60, 61

IT 7722-84-1, **Hydrogen peroxide**, reactions  
(in **prodn.** of chlorine dioxide by chlorate redn. with chlorides)

L66 ANSWER 8 OF 28 HCA COPYRIGHT 2003 ACS on STN

124:162480 Method and apparatus for cleaning a workpiece. Maekawa, Toshiro; Ono, Koji; Okada, Motoaki; Takahashi, Tamami; Mishima, Shiro; Koderu, Masako; Shigeta, Atsushi; Aoki, Riichiro; Kouno, Giusuke (Ebara Corp., Japan). Eur. Pat. Appl. EP 692318 A1 19960117, 20 pp. DESIGNATED STATES: R: DE, FR, GB, NL. (English). CODEN: EPXXDW. APPLICATION: EP 1995-107411 19950515. PRIORITY: JP 1994-168767 19940628.

AB A semiconductor wafer is scrubbed by a cleaning member made primarily of polyurethane and having micropores in a surface contacting the semiconductor wafer. The micropores have an av. diam. of 10-200 .mu.m. The cleaning member may be made of either polyurethane foam or nonwoven fabric composed of fibers bound together by urethane resin. Particles that are strongly attached to the surface of a substrate such as a semiconductor wafer can be

easily removed by scrubbing. At the same time a substrate is cleaned, surface irregularities and **cryst.** protrusions on the surface of the substrate (esp. a semiconductor wafer) can be **scraped** off to adjust the surface roughness of the wafer to a desired degree for making the wafer surface flat.

IC ICM B08B001-04  
ICS B24B037-04; H01L021-306; H01L021-00; B24D003-32  
CC 76-3 (Electric Phenomena)  
IT 7647-01-0, Hydrogen chloride, processes 7664-41-7, Ammonia, processes **7722-84-1, Hydrogen peroxide**, processes  
(cleaning of semiconductor wafers by solns. contg.)

L66 ANSWER 9 OF 28 HCA COPYRIGHT 2003 ACS on STN

124:126323 ~~Release of major ions and hydrogen peroxide from homogeneous, melting snow.~~ Herrmann, Reimer, Kranz, Jutta (Universitat Bayreuth, Germany). Nordic Hydrology, 26(4-5), 359-68 (English) 1995. CODEN: NOHYBB. ISSN: 0029-1277. Publisher: Nordic Association for Hydrology.

AB We undertook 2 lab. expts. to study the change of concns. and the preferential elution of major ions and **H2O2** during **melting** of a homogeneous snow pack. The elution sequence was as follows: alkali metals, alk.-earth metals and protons > anions > **NH4+** > **H2O2**. We explain the late elution of **Cl-**, **NH4+** and **H2O2** by high ice/water partition coeffs. and a more even distribution of the two ions as well as of **H2O2** within the ice **crystal** due to formation processes of snow particles in the atm.

CC 61-1 (Water)

ST **hydrogen peroxide melting** snow chem

IT Alkali metals, occurrence

Alkaline earth metals

Chlorides, occurrence

(release of major ions and **hydrogen peroxide** from homogeneous **melting** snow)

IT Waters, natural

(snowmelt, release of major ions and **hydrogen peroxide** from homogeneous **melting** snow)

IT **7722-84-1, Hydrogen peroxide**, occurrence

14798-03-9, Ammonium, occurrence

(release of major ions and **hydrogen peroxide** from homogeneous **melting** snow)

L66 ANSWER 10 OF 28 HCA COPYRIGHT 2003 ACS on STN

121:110 flow cytometric detection of **hydrogen peroxide**

**production** induced by doxorubicin in cancer cells. Ubezio, Paolo; Civoli, Francesca (Ist. Ricerche Farmacologiche "Mario Negri", Milan, Italy). Free Radical Biology & Medicine, 16(4), 509-16 (English) 1994. CODEN: FRBMEH. ISSN: 0891-5849.

AB 2',7'-Dichlorofluorescein diacetate (DCFH-DA) has been previously used to study the oxidative burst of neutrophils induced by different stimuli. The method is based on the fact that DCFH-DA

diffuses through the cell membrane and it is hydrolyzed by intracellular esterases to DCFH, which remains trapped within the cells. DCFH, a nonfluorescent compd., is able to react with free radical **products**, particularly with **hydrogen peroxide**, and to **generate** the fluorescent 2',7'-dichlorofluorescein (DCF). By flow cytometric detection of DCF fluorescence, an indirect measure of reactive oxygen species prodn. in single cells may be obtained. Using a modified procedure to load cells of the human colon adenocarcinoma cell line LoVo with DCFH-DA, a significant fluorescence increase above the basal fluorescence level has been detected after treatment with doxorubicin doses as low as 0.4  $\mu$ M. This increase is not detectable when the cells are preloaded with catalase, using a **scraping** method, and it is not due to doxorubicin own fluorescence. These expts. prove that the increase of DCF fluorescence intensity obsd. during doxorubicin treatment is not due to tech. artifacts but it is attributable to free radicals produced in the cells by the drug.

- CC 1-1 (Pharmacology)  
 Section cross-reference(s): 9
- ST cancer **hydrogen peroxide formation**  
 doxorubicin; flow cytometry **hydrogen peroxide formation**
- IT Neoplasm, metabolism  
 (**hydrogen peroxide formation** by,  
 after induction by doxorubicin, flow cytometry in detn. of)
- IT 7722-84-1, **Hydrogen peroxide**, analysis  
 (detn. of **formation** of, in cancer cells after induction  
 by doxorubicin, flow cytometry in)
- IT 23214-92-8, Doxorubicin  
 (**hydrogen peroxide formation**  
 induced by, in cancer cells, flow cytometry in detn. of)
- IT 4091-99-0, 2',7'-Dichlorofluorescein diacetate  
 (in flow cytometric detection of **hydrogen peroxide prodn.** induced by doxorubicin in  
 cancer cells)

L66 ANSWER 11 OF 28 HCA COPYRIGHT 2003 ACS on STN  
 120:9517 Study on polythiophenylene/polyether-polysulfone blend prepared by coprecipitation technique. Yang, Chen; Zeng, Hanmin (Mater. Sci. Inst., Zhongshan Univ., Canton, 510275, Peop. Rep. China). Gaofenzi Cailiao Kexue Yu Gongcheng, 8(4), 81-7 (Chinese) 1992. CODEN: GCKGEI. ISSN: 1000-7555.

AB The structure, melting, **crystn.**, and phase sepn. behaviors of polythiophenylene (I)/polyether-polysulfone (PES) blends prepd. by copptn. in  $\alpha$ -chloronaphthalene-DMSO mixt. using EtOH as pptn. agent were studied by DSC, IR, and wide-angle x-ray spectra. The **cryst.** peak of the DSC curves of the blends became broader and shifted to lower temp. direction with increasing PES content. The heat of **fusion** of untreated and H2O2-treated I was 48.0 J/g and 41.8 J/g and the heat of **crystn.** was 47.3 J/g and 33.8 J/g, resp., indicating that H2O2

treatment changed the mol. structure of I. H2O2-treated I had stronger interaction with PES compared with the untreated I. The interaction between I and PES was dependent on the compn. ratio of the blend. The cooling rate was the key factor controlling the **crystn.** and phase sepn. of the blends.

- CC 37-5 (Plastics Manufacture and Processing)
- ST polythiophenylene polyether polysulfone blend property; melting  
polythiophenylene polyether polysulfone blend; **crystn**  
polythiophenylene polyether polysulfone blend; phase sepn  
polythiophenylene polyether polysulfone
- IT Heat of **crystallization**  
Heat of fusion and Heat of freezing  
(of polyether-polysulfone blends with **hydrogen peroxide**-treated and untreated polythiophenylenes)
- IT **Crystallization**  
(of polyether-polysulfone blends with **hydrogen peroxide**-treated and untreated polythiophenylenes, compn. effect on)
- IT Polythiophenylenes  
(polyether-polysulfone blends, structure, melting, **crystn** and phase sepn. behaviors of)
- IT Polysulfones, properties  
(polyether-, blends with **hydrogen peroxide**-treated and untreated polythiophenylenes, structure, melting, **crystn.** and phase sepn. behaviors of)
- IT Polyethers, properties  
(polysulfone-, blends with **hydrogen peroxide**-treated and untreated polythiophenylenes, structure, melting, **crystn.** and phase sepn. behaviors of)
- IT 25667-42-9, Poly(oxy-p-phenylenesulfonyl-p-phenylene)  
(blends with **hydrogen peroxide**-treated and untreated polythiophenylenes, structure, melting, **crystn** and phase sepn. behaviors of)
- IT 25212-74-2, Polythiophenylene  
(polyether-polysulfone blends, structure, melting, **crystn** and phase sepn. behaviors of)
- IT 7722-84-1, **Hydrogen peroxide**, uses  
(polythiophenylene treatment with, properties in blends with polyether-polysulfones in relation to)

L66 ANSWER 12 OF 28 HCA COPYRIGHT 2003 ACS on STN

114:91616 Manufacturing process of light-emitting diode. Okabe, Tadatoshi (Shinnippon Musen Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 02254773 A2 19901015 Heisei, 4 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 1989-75025 19890329.

AB The title process for making an LED enhancing upward emission from the pn-junction surface comprises the steps of: forming a pn-junction using dopants by LPE of a ternary Al<sub>x</sub>Ga<sub>1-x</sub>As single **crystal** with an Al concn. gradient resulting from Al segregation coeff.; removing the epitaxy substrate by chem. etching; forming electrodes on both end surfaces of the **crystal** and making chips by dicing or **scrape**-breaking; etching the

chip to a mesa shape by a soln. having an Al concn.-dependent etching rate, e.g.  $\text{H}_2\text{O}_2:\text{NH}_4\text{OH} = 30:1$ ; and mounting the mesa chip into a package with a larger base of the mesa up. Alternatively, a binary GaAs is used, where in the mesa shape is formed by a depth-dependent diffusion gradient in the etching sol. The LED gives 30% more light output than the conventional one.

IC ICM H01L033-00

CC 73-10 (Optical, Electron, and Mass Spectroscopy and Other Related Properties)

Section cross-reference(s): 76

L66 ANSWER 13 OF 28 HCA COPYRIGHT 2003 ACS on STN

99:40496 Separation membrane washing. (Kurita Kogyo Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 58020205 A2 19830205 Showa, 5 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 1981-119302 19810731.

AB Washing solns. for contaminated membranes, e.g., in filtration, ultrafiltrations (UF), reverse osmosis, and microfiltration, consist of oxalic acid (0.01-1%) and surfactant (>0.3%) as the washing liq.,  $(\text{NaPO}_3)_6$  for preventing Ca and Fe salt formation, and  $\text{H}_2\text{O}_2$  as the exfoliation agent and are used together with a mech. means with movement of elastic matter. Thus, a contaminated module of tubular polysulfonate UF membranes was washed to the accumulated residual matter of  $\text{CaO}$  2.2,  $\text{Fe}_2\text{O}_3$  1.1,  $\text{MnO}$  0.9,  $\text{Al}_2\text{O}_3$  5.4,  $\text{P}_2\text{O}_5$  1.7, and insol. acids 16.7% with a trace of  $\text{MgO}$  from oil-contg.-waste water filtration, when the rate of the filtration was reduced to <0.01 m/day. The rate was recovered to 0.03-0.05 m/day after the mech. **scraping** 30 times with a sponge ball. Then, it was recovered further to 0.55 m/day after washing with a mixt. of oxalic acid and Emulgen 810 followed by addnl. mech. **scraping** for 30 times.

IC B01D013-00; B01D031-00

CC 48-1 (Unit Operations and Processes)

Section cross-reference(s): 66

IT Filtration

(deposit removal from membranes in, by **scraping** and washing)

IT Filtration

(micro-, deposit removal from membranes in, by **scraping** and washing)

IT Filtration

(ultra-, deposit removal from membranes in, by **scraping** and washing)

L66 ANSWER 14 OF 28 HCA COPYRIGHT 2003 ACS on STN

96:128580 Foamed gypsum molded articles. Saito, Masumi; Hirai, Eiichi; Endo, Masao; Nishino, Toru (Kurashiki Spinning Co., Ltd., Japan). Eur. Pat. Appl. EP 40399 A1 19811125, 25 pp. DESIGNATED STATES: R: DE, FR, GB. (English). CODEN: EPXXDW. APPLICATION: EP 1981-103688 19810513. PRIORITY: JP 1980-65060 19800515.

AB Foamed sound insulators for structural materials in buildings are prepd. from gypsum with interconnected cells **formed** by  $\text{H}_2\text{O}_2$  decompn. by a Co compd. produced by pretreating

water-sol. Co salts with H<sub>2</sub>O<sub>2</sub> in the presence of alkalies. Thus, 6% H<sub>2</sub>O<sub>2</sub> 0.5 and an aq. soln. of CoSO<sub>4</sub>·7H<sub>2</sub>O 0.1 and KOH 2 are stirred with gypsum 100 in water 57 and then with 6% H<sub>2</sub>O<sub>2</sub> 5.5 and polyacrylic ester emulsion wt. parts. During or immediately after expansion, the resultant slurry is cast in a gypsum board mold, held 15 min, demolded, aged .apprx.1 h, and dried 6 h at 45.degree.. The surface is **scraped** with sand to expose the porous surface and the sound absorbing properties are detd. On vertical incidence of 250, 400, 630, 1000, 1600, and 2000 Hz on the gypsum board, the resp. sound absorption is 11, 21, 38, 50, 57, and 56%.

IC C04B021-02; C04B013-00  
CC 58-4 (Cement, Concrete, and Related Building Materials)

L66 ANSWER 15 OF 28 HCA COPYRIGHT 2003 ACS on STN

82:113694 Hydrogen peroxide. Pneva, E. Ya.; Selyutina, L. P.; Makkaveeva, L. E.; Krokhamlyuk, A. E.; Dmitrieva, L. Ya.; Glazman, T. P.; Shkurkin, T. G.; Speer, T. V.; Klinaev, Yu. M.; et al. Ger. Offen. DE 2326804 19741212, 24 pp. (German). CODEN: GWXXBX.  
APPLICATION: DE 1973-2326804 19730525.

AB H<sub>2</sub>O<sub>2</sub> is produced by oxidizing a secondary alc.  
with air at 70-160.degree. and 1-70 atm in a multiple-zone reacto in a **countercurrent** operation so that the ratio of O<sub>2</sub> concn. of the gas leaving the individual zones to the concn. of **H<sub>2</sub>O<sub>2</sub> formed** in each zone is >1.3. The process is accelerated by the addn. of 0.1-4 wt.% of an ether to the reaction mixt. and the product is purified with direct steam, pptg. the acid formed as its salt. Thus, Me<sub>2</sub>CHOH contg. 160 mg NaH<sub>2</sub>PO<sub>4</sub>/l. is introduced continuously into the upper section of a 3-zone reactor at 25 l./hr, while the lower sections receive the condensate at 12 l./hr. Air is blown in at 133 m<sup>3</sup> hr<sup>-1</sup>m<sup>-2</sup> of the app. at 10 atm and 115-127.degree.. The H<sub>2</sub>O<sub>2</sub> yield is 97% and the concn. of AcOH 0.19 wt.%. The reaction mixt. is purified with direct steam. The escaping H<sub>2</sub>O vapor is contacted at 150 torr and 60.degree. with an aq. NaOAc soln., maintained with NaOH at a pH of 12, and the pure **cryst.** NaOAc filtered off. The process can be accelerated by a factor of 1.5 by adding 2% iso-Pr<sub>2</sub>O to the initial alc.

IC C01B  
CC 49-8 (Industrial Inorganic Chemicals)  
ST **hydrogen peroxide prepn; alc oxidn**  
IT 108-20-3

(accelerator, in **hydrogen peroxide**  
**manuf.**)

IT 7722-84-1P, **preparation**  
(from isopropyl alc., by oxidn., isopropyl ether in)

IT 67-63-0, **reactions**  
(oxidn. of, in **hydrogen peroxide**  
**manuf.**)

L66 ANSWER 16 OF 28 HCA COPYRIGHT 2003 ACS on STN

63:43271 Original Reference No. 63:7756g-h,7757a-b Semiconductor. (N. V. Philips' Gloeilampenfabriken). BE 643479 19640806, 36 pp.  
(Unavailable). PRIORITY: NL 19630329.

AB A strip of n-type semiconductor (e.g. Ge), of .apprx.0.5 ohm/cm. resistivity and 10 .times. 10 mm. .times. 1 .mu. size, contg. Sb (3 .times. 10<sup>16</sup> atoms/cc.) is heated 2 hrs. at 800.degree. under an O atm. with an In-Ge alloy (In = 60 at. %). A p-type layer is formed by the slow diffusion of the In in the semiconductor. To obtain 100 n-p-n, very high-frequency transistors, 100 Al disks (60 .mu. in diam., 0.4 .mu. thick, and .apprx.0.9 mm. apart) are evapd. on the upper side of the strip. The disks have a semicircular cavity of 0.6 .mu. radius and the same center as the disk. The Al is alloyed to the plate by heating 1 min. at 730.degree. in a H atm. The vaporization of the Al is preceded by the application of In (6% of the Al) to obtain a more uniform Al-Ge alloy. The opposite side of the strip is **scraped** in a bath of HF (50%) 10, HNO<sub>2</sub> (65%) 14, H<sub>2</sub>O 1, and EtOH 0.5 part to a depth of .apprx.5 .mu. to remove the p-layer. The other surfaces are protected with wax. The plate is then heated 9 min. at 650.degree. in a H atm. and As vapor is allowed from a furnace where it was heated to 440.degree.. The As diffuses into the semiconductor, forming a surface n-type layer (.apprx.0.6 .mu. thick), discontinued at the Al alloy regions; on cooling, the Al alloy sep. into 2 layers. The lower one is a recrystd. p-type and the upper layer acts as a base connection. The portion of n-type (As) layer, in the cavity of the Al disk, constitutes the emitter zone. A section surrounding the Al disk is **scraped** with the bath indicated above. Wax masking is used to protect the other parts. The wax is removed and the plate is broken into 100 transistors. Au wires are connected to the Al disk and to the layer in the cavity and a gilded fernico is soldered at 500.degree. to the opposite side. Metal residues are removed from the surface with 10% H<sub>2</sub>O<sub>2</sub> at 40.degree.. These transistors have exceptional amplification properties at very high frequencies.

CC 9 (Electric and Magnetic Phenomena)

L66 ANSWER 17 OF 28 HCA COPYRIGHT 2003 ACS on STN  
57:53123 Original Reference No. 57:10580b-c Oxidation-reduction analysis of cryogenically stable products of dissociated water vapor. Gladney, Henry M.; Garvin, David (Princeton Univ., Princeton, NJ). Journal of Physical Chemistry, 66, 1500-2 (Unavailable) 1962. CODEN: JPCHAX. ISSN: 0022-3654.

AB Deposits having similar characteristics are obtained by trapping at 77.degree.K. the products from discharged H<sub>2</sub>O vapor or HO<sub>2</sub>, or from the reaction of H atoms with O or O<sub>3</sub>. The deposits are partly **cryst.**, **recrystallize** with evolution of O on warming, and form concd. solns. of H<sub>2</sub>O<sub>2</sub> on **melting**. From oxidn. and redn. analyses of the solid and melted materials, it was shown that a species exists which is stable only at low temps. This low temp. species undergoes typical chem. reactions.

CC 6 (Phase Equilibriums, Chemical Equilibriums, and Solutions)

IT Electric discharge

(dissozn. of H<sub>2</sub>O<sub>2</sub> and water vapor in, cryogenically stable products from)

IT 7722-84-1, Hydrogen peroxide

(dissozn. of, in elec. discharge, cryogenically stable products from)

L66 ANSWER 18 OF 28 HCA COPYRIGHT 2003 ACS on STN

55:27494 Original Reference No. 55:5342e-i,5343a-c Preparation of organic compounds labeled with S35. A convenient method for obtaining labeled isothiocyanates. Iliceto, Antonio; D'Angeli, Ferruccio (Univ. Padua, Italy). Gazzetta Chimica Italiana, 89, 1950-5 (Unavailable) 1959. CODEN: GCITA9. ISSN: 0016-5603.

AB cf. CA 51, 3458e. Alk. sulfides, RS35H (I), reacted with a large excess of isothiocyanate, RNCS (II), to give labeled dithiocarbamates RHNCSS35H (III), with uniform distribution of radioactivity between II and III due to isotopic interchange between II and III. RCSNH2 and RCS2H underwent isotopic interchange at a measurable rate, contrary to (H2N)CS, for which there was no appreciable interchange with S35H-. Redn. of BaS35O4 in a stream of H at 950.degree., double exchange between BaS35 and Na2SO4 and 0.5-2.0 ml. aq. Na2S35 dild. extensively with aq. or alc. NaHS gave solns. of NaS35H of the desired concn. II, PhNHCSMe (m. 76-77.degree.), PhCSNH2, and CS(SCH2CO2Na)2 (IV), m. 171-2.degree. (H2O), were prepd. according to the literature. The S35 of org. compds. transformed into S35O4 ion by oxidn. with alk. **H2O2** or alk. oxidn. **fusion** was pptd. as BaS35O4 and the radioactivity measured in thick layers under standard conditions. NaSH (0.02 mole) in 5 ml. H2O treated with 0.02 mole MeNCS and 2.5 ml. soln. concd. (N atm.) in vacuo yielded 55% MeHNCS2Na. The remaining soln. (2.5 ml.) dild. to 0.1N and treated slowly with stirring at 0.degree. with 0.1N iodine yielded 68% (MeNHCS2)2, m. 100-1.degree.. MeNCS (3.65 g.) in 9 ml. alc. and 1 ml. 2N NaS35H in H2O kept 30 min. at 20.degree., treated with 10 ml. H2O and 15 ml. Et2O, the Et2O layer washed 4 times with 10 ml. H2O, the dried (Na2SO4)Et2O divided into 2 equal portions, one evapd. to give 1.2 g. active II (R = Me), b. 115-17.degree., m. 34-5.degree., activity 980 counts/min. (c./m.), and the other concd., treated with NH4OH and filtered gave (MeNH)2 CS35, m. 119-20.degree., activity 950 c./m. Oxidn. of the aq. soln. with iodine gave (MeNHCS352)2, m. 100-1.degree., activity 1010 c./m. Similarly, 0.05 mole II (R = Et) gave 3 g. active II (R = Et), m. 129-31.degree., activity 1720 c./m. and (EtNHCS352)2, m. 69-71.degree., activity 1750 c./m. Under the same conditions, analogous reactions between II (R = Me2CH, PhCH2) and I (R = Na) and addn. of concd. NH4OH to the alc. aq. mixt. gave the corresponding thioureas with the expected activities. Active II (R = Me) (0.5 mole) treated at 20.degree. with 0.25 mole MeNHCS2Na in 90% alc., 2 ml. soln. dild. with 3 ml. H2O and 3 ml. Et2O, the layers washed 3 times with 2 ml. Et2O and H2O, the 2 combined Et2O and H2O solns. (contg. MeNCS and MeNHCS2Na, resp.) oxidized sep. with alk. **H2O2**, and the fractional exchange detd. at intervals gave 0.10, 0.35, 0.48, 0.61, 0.72 in 0, 5, 10, 15, 20 min., resp., a half-life change of 13 min. The exchange between MeNHCS2- and S35H- was examd. in 0.5M NaS35H and 0.25M MeNHCS2Na, the soln. treated with N HClO4, extd. with Et2O, the ext. washed with aq. AgNO3 to eliminate the H2S, the CS2 oxidized with



alk. **H2O2**, and pptd. as BaSO<sub>4</sub>. At 20.degree., the half-life exchange exceeded 100 hrs. In tests of exchange between thioamides or thio esters with NaS<sub>3</sub>SH, the org. compd. was used in 3-6 fold excess and the products carefully purified after sepn. by repeated **crystn.** or pptn. NaS<sub>3</sub>SH (0.1 mole) and 0.3 mole PhCSNH<sub>2</sub> in 98% MeOH was acidified with AcOH, the H<sub>2</sub>S removed in a stream of N, the MeOH evapd., and the PhNHCS<sub>3</sub>Me **recrystd.** 3 times from H<sub>2</sub>O prior to oxidn. with alk. **H2O2**. At 40.degree., exchanges of 14 and 21% were observed in 3 and 6 hrs., resp., a half-life exchange of about 20 hrs. NaS<sub>3</sub>SH (0.1 mole) and 0.6 mole PhNHCSMe in 80% alc. treated with 5 vols. H<sub>2</sub>O, the H<sub>2</sub>O-washed ppt. taken up in alc., and repptd. with H<sub>2</sub>O gave PhNHCS<sub>3</sub>Me, oxidized with alk. **H2O2**. At 60.degree. 26 and 48% exchange was noted in 6 and 12 hrs., resp., a half-life exchange of 13 hrs. NaS<sub>3</sub>SH (0.05 mole) and 0.25 mole IV in H<sub>2</sub>O treated with concd. HCl and the product repptd. from alk. soln. followed by alk. **H2O2** oxidn. showed 6 and 11% change after 1.5 and 3 hrs., indicating a half-life change of 16 hrs.

CC 10B (Organic Chemistry: Aliphatic Compounds)

L66 ANSWER 19 OF 28 HCA COPYRIGHT 2003 ACS on STN

54:62296 ~~Original Reference No. 54:11986h-i,11987a-i,11988a-f~~

~~Alternaric acid. II. Oxidation. Bartels-Kelth, J. R. (Imp. Chem. Inds. Ltd., Welwyn, UK). Journal of the Chemical Society, Abstracts 860-6 (Unavailable) 1960. CODEN: JCSAAZ. ISSN: 0590-9791.~~

AB cf. C.A. 47, 8685c. Alternaric acid (I) was oxidized by alk. **H2O2** to 7,8-dihydroxy-9-methyl-3-methyleneundec-5-ene-1,7-dicarboxylic acid (II). I (5.02 g.), 77.6 ml. N NaOH, and 150 ml. 20-vol. **H2O2** made up to 850 ml. with H<sub>2</sub>O, left 3 days at room temp. then 6 days with 100 ml. more **H2O2**, acidified, extd. with Et<sub>2</sub>O, washed with 25% NaCl, dried, and evapd. gave 3.2 g. II, leaflets, m. 95.degree. (EtOAc-ligroine), [ $\alpha$ ]D<sub>21</sub> 43.5.degree. (c 2.01, Me<sub>2</sub>CO),  $\lambda$ . 210 m $\mu$ .,  $\epsilon$ . 4560,  $\nu$ . 3300-3400, 2500, 1718, 1701, 1684, 1669, and 1645 cm.<sup>-1</sup> Hydrogenation of I in AcOH with Pd gave an uptake of H corresponding to 1.9 double bonds. II (299 mg.) methylated with Et<sub>2</sub>O-CH<sub>2</sub>N<sub>2</sub> and the product chromatographed on Al<sub>2</sub>O<sub>3</sub> gave 252 mg. Me ester (III), m. 41-3.5.degree. (Et<sub>2</sub>O-ligroine), [ $\alpha$ ]D<sub>19</sub> 48.0.degree. (c 2.01, alc.),  $\lambda$ . 210 m $\mu$ .,  $\epsilon$ . 4440,  $\nu$ . 3515, 1720, and 1642 cm.<sup>-1</sup> III (198 mg.) with 4 ml. Ac<sub>2</sub>O and 5.4 ml. C<sub>5</sub>H<sub>5</sub>N 20 hrs. at room temp. gave 201 mg. product, which chromatographed on Al<sub>2</sub>O<sub>3</sub> gave 89 mg. di-Me 8-acetoxy-7-hydroxy-9-methyl-3-methyleneundec-5-ene-1,7-dicarboxylate, b<sub>10-4</sub> 70-80.degree.,  $\nu$ . 3510, 1743, and 1650 cm.<sup>-1</sup> II (100 mg.) in 10 ml. Me<sub>2</sub>CO contg. 0.05 ml. concd. H<sub>2</sub>SO<sub>4</sub> left 18 hrs. at room temp., the mixt. shaken 0.5 hr. with 0.63 g. NaHCO<sub>3</sub>, cooled to 0.degree., shaken with 20 ml. H<sub>2</sub>O and 20 ml. Et<sub>2</sub>O, the aq. layer acidified at 0.degree., extd. with Et<sub>2</sub>O, evapd., the 120 mg. sirup in 0.85 ml. N NaOH adjusted to pH 3, left 1 hr. at 0.degree. with 135 mg. S-benzylthiuronium chloride in 1 ml. H<sub>2</sub>O, and the solid collected gave 184 mg. bis(S-benzylthiuronium) 7,8-isopropylidenedioxy-9-methyl-3-methylene-5-undecene-1,7-dicarboxylate, m. 173.5-4.5.degree. (alc.). III (532 mg.) in 50 ml.

Me<sub>2</sub>CO contg. 0.25 ml. concd. H<sub>2</sub>SO<sub>4</sub> left 18 hrs. at room temp. and the crude product chromatographed on Al<sub>2</sub>O<sub>3</sub> gave 530 mg. isopropylidene deriv., [ $\alpha$ ]D<sub>20</sub> 33.degree. (c 4.53 alc.). Mol. distn. at 60-75.degree./0.001 mm. gave the anal. sample,  $\lambda$ . 210 m. $\mu$ .,  $\epsilon$ . 3248,  $\nu$ . 1743, and 1651 cm.<sup>-1</sup> I (2 g.) oxidized as above and the acidified mixt. distd., the distillate made alk., satd. with CO<sub>2</sub>, filtered, distd., and treated with 2,4-dinitrophenylhydrazine-HCl gave a complex mixt. of 2,4-dinitrophenylhydrazones. This mixt. was also obtained in an expt. in which the H<sub>2</sub>O<sub>2</sub> was omitted. Fractional **crystn.** gave acetone 2,4-dinitrophenylhydrazone, m. 123-5.degree.. The residue from the 2nd distn. gave 228 mg. Ba(HCO<sub>2</sub>)<sub>2</sub>, which was converted into S-benzylthiuronium formate, m. 150.5-1.5.degree.. II (152 mg.) in 1 ml. MeOH treated at room temp. with 4.5 ml. 0.12M HIO<sub>4</sub>, volatile product removed with a current of N, treated 45 min. at 5.degree. with 2,4-dinitrophenylhydrazine in 4N HCl, the ppt. collected, chromatographed on Al<sub>2</sub>O<sub>3</sub>, and eluted with C<sub>6</sub>H<sub>6</sub> gave (+)- $\alpha$ -methylbutyraldehyde 2,4-dinitrophenylhydrazone (IIIa), m. 134-4.5.degree. (MeOH), [ $\alpha$ ]D<sub>18</sub> 32.6.degree. (c 2.5, CHCl<sub>3</sub>). III (323 mg.), 6.3 ml. 0.17M HIO<sub>4</sub>, and alc. contg. 6 ml. 5% EtOAc kept 1 day in the dark, distd. at 25.degree./25 mm., after 1 hr. the distillate added to 175 ml. 0.2% aq. dimedon contg. 2 drops AcOH, after 5 days the product collected, and **recrystd.** gave 172 mg. dimedon deriv. of (+)- $\alpha$ -methylbutyraldehyde (IV), m. 134-5.degree. (aq. MeOH), [ $\alpha$ ]D<sub>20</sub> 2.degree. (c 1.88, alc.). In a 2nd expt. 1.05 g. III gave a distillate, which refluxed under N with 0.4 g. semicarbazide-HCl and 0.6 g. NaOAc.3H<sub>2</sub>O, evapd. 1 hr., and dild. with H<sub>2</sub>O gave 240 mg. semicarbazone of IV, m. 113-15.degree. (aq. alc.). The residue from the distn. extd. with Et<sub>2</sub>O, the exts. washed with 9% aq. NaHCO<sub>3</sub> and 10% NaCl, distd., and treated with 2,4-dinitrophenylhydrazine gave 102 mg. 2,4-dinitrophenylhydrazone of di-Me 6-methylene-2-oxonon-3-enedioate (V), m. 149-50.degree. (EtOAc-ligroine),  $\lambda$ . 217, 253, 257, 375 m. $\mu$ .,  $\epsilon$ . 20,510, 11,880, 11,570, 23,810. III (101 mg.) in 5 ml. C<sub>6</sub>H<sub>8</sub> treated 1.5 hrs. with 154 mg. Pb(OAc)<sub>4</sub> and the product treated with 2,4-dinitrophenylhydrazine in HCl gave 18 mg. IIIa. The residue from the distn. similarly treated gave 48 mg. V. III (492 mg.) in 25 ml. CHCl<sub>3</sub> shaken with 5 g. active MnO<sub>2</sub> 23 hrs. at room temp. gave 55 mg. oil,  $\lambda$ . 233 and 302 m. $\mu$ ., E<sub>1cm</sub>.1% 255, 212, and 204 mg. oil,  $\lambda$ . 233 m. $\mu$ ., E<sub>1cm</sub>.1% 253. The latter product (46 mg.) with 2,4-dinitrophenylhydrazine gave 14 mg. V. Di-Me 7,8-isopropylidenedioxy-9-methyl-3-methylene-5-undecene-1,7-dicarboxylate (1.047 g.) in 50 ml. Me<sub>2</sub>CO treated 1 hr. at 24.degree. with 4 g. KMnO<sub>4</sub>, after 3 hrs. the mixt. cooled to 0.degree., decolorized by H<sub>2</sub>SO<sub>3</sub>, and extd. with Et<sub>2</sub>O gave 631 mg. sirup, converted into S-benzylthiuronium Me 2,2-dimethyl-5-sec-butyl-1,3-dioxolane-4,4-dicarboxylate (VI), m. 146.degree., [ $\alpha$ ]D<sub>23</sub> 64.degree. (c 1, alc.). VI (1.37 mg.) shaken at 0.degree. with 10 ml. Et<sub>2</sub>O and 5 ml. 3N H<sub>2</sub>SO<sub>4</sub>, the aq. layer extd. with Et<sub>2</sub>O, and evapd. gave Me H 2,2-dimethyl-5-sec-butyl-1,3-dioxolane-4,4-dicarboxylate (VII), viscous sirup. VII (190 mg.) in 12 ml. 0.4N KOH left 20 hrs. at room temp., washed with Et<sub>2</sub>O, cooled, acidified

with 3N H<sub>2</sub>SO<sub>4</sub>, and extd. with Et<sub>2</sub>O gave 171 mg. diacid, m. 88.5-94.degree. (CH<sub>2</sub>Cl<sub>2</sub>-ligroine), .nu. 3100-2500, 1728, and 1715 cm.<sup>-1</sup>; Ag salt decompd. above 150.degree.. II (143 mg.) in 15 ml. AcOH treated 14 min. at 19.degree. with a stream of O<sub>3</sub>-O<sub>2</sub>, Mg powder and 70 ml. H<sub>2</sub>O added, the mixt. distd., and the distillate left 6 days with 150 ml. 0.2% dimedon gave 71 mg. formaldehyde dimedon deriv. (VIII), m. 189-91.degree. (MeOH). This represents a 58% yield of HCHO. Ozonized air passes into 144 mg. II in 12 ml. MeOAc 14 min. at -50.degree., the ozonide decompd. with H<sub>2</sub>O, and steam distd. gave after 3 days with dimedon soln. 58 mg. VIII. The residue from the steam distn. extd. continuously 24 hrs. with Et<sub>2</sub>O and evapd. gave 111 mg. residue. The 9 mg. of undissolved solid gave 5.8 mg. succinic acid, m. 187.degree.. Evapn. of the CH<sub>2</sub>Cl<sub>2</sub> soln. gave an oil which refluxed 1.5 hr. with 0.20 g. 2,4-dinitrophenylhydrazine and concd. HCl gave 32 mg. Me levulinate 2,4-dinitrophenylhydrazone (VIIIa), m. 140.degree. (C<sub>6</sub>H<sub>6</sub>). II (30.5 mg.) in 10 ml. contg. 0.3 ml. 4N Na<sub>2</sub>CO<sub>3</sub> and 2 ml. N NaOH treated 1 min. with 0.56 g. NaIO<sub>4</sub> and 1 ml. 0.034M KMnO<sub>4</sub> in 20 ml. H<sub>2</sub>O, 2 ml. N HCl added, after 3 hrs. a soln. of 2 g. As<sub>2</sub>O<sub>3</sub> in 8 ml. 3N NaOH added followed by 15 ml. 3N H<sub>2</sub>SO<sub>4</sub>, and the mixt. steam distd. gave HCHO, isolated as the dimedon deriv. in 7.3-mg. yield. In a parallel expt. titrimetric analysis showed that II consumed 9 atoms O/mol. in 1.5 hrs., and thereafter oxidn. was slow, 10.7 atoms being consumed in 18 hrs. N was passed successively through a vessel contg. 1.59 g. Pb(OAc)<sub>4</sub>, a trap contg. 160 ml. 0.3% 2,4-dinitrophenylhydrazine in 4N HCl, an empty trap at -80.degree., and 2 traps contg. in all 100 ml. 0.147N Ba(OH)<sub>2</sub>, 479 mg. II in 25 ml. 60% aq. AcOH added, after 2 hrs. the contents of the Ba(OH)<sub>2</sub> traps made up to 200 ml. with H<sub>2</sub>O. This soln. (20 ml.) neutralized 11.6 ml. 0.100N HCl, equiv. to formation of 0.98 mole CO<sub>2</sub>/mole II. The mixt. distd. at 20.degree./2 mm. into the cold trap and the distillate added to the contents of the 2,4-dinitrophenylhydrazine trap gave 227 mg. IIIa. The residue from the above distn. treated with 2 ml. 3N H<sub>2</sub>SO<sub>4</sub>, extd. with EtOAc, the combined exts. washed with 25% NaCl, dried, and evapd. gave 57 mg. 5-methylene-2-octenedioic acid (IX), prisms, m. 120-2.degree. (Me<sub>2</sub>CO-CCl<sub>4</sub>), [.alpha.]<sub>D</sub><sub>20</sub> 1.degree. (c 2, Me<sub>2</sub>CO), .lambda. 208 m.mu., .epsilon. 12,860, .nu. 1700, 1685, and 1648 cm.<sup>-1</sup> IX sublimed at 150-80.degree./15 mm. with only slight decompn. and was recovered after refluxing 1 hr. with AcCl. The crude nonvolatile oxidn. product (1.18 g.) subjected to 100 transfers in a **countercurrent** app. with 4:4:1 AcOH-C<sub>6</sub>H<sub>6</sub>-H<sub>2</sub>O, and the contents of tubes 25-41 evapd. at 20.degree./2 mm. gave 464 mg. IX. IX (118 mg.) in 10 ml. EtOAc hydrogenated 0.5 hr. with 28 mg. PtO<sub>2</sub> gave 112 mg. (+)-.alpha.-methylsuberic acid (X), prisms, m. 64.5-7.0.degree. (C<sub>6</sub>H<sub>6</sub>-ligroine). X (112 mg.) in 1.2 ml. N NaOH treated 1 hr. with 1 ml. H<sub>2</sub>O and 180 mg. thorium nitrate-4H<sub>2</sub>O, the ppt. collected, washed, and dried. gave 194 mg salt. The salt heated 0.5 hr. at 340-50.degree./1 mm., then heated slowly to 390.degree., and the cold trap contents treated with semicarbazide gave 49 mg. 4-methylcycloheptanone semicarbazone, prisms, m. 153-5.degree. (alc.). O<sub>3</sub>-O<sub>2</sub> passed through 62 mg. IX in 8 ml. MeOAc

14 min. at -50.degree., 120 ml. H<sub>2</sub>O added, and the mixt. distd. gave from the aq. distillate 67.5% HCHO dimedon deriv. The residue from the distn. extd. 20 hrs. with Et<sub>2</sub>O and evapd. gave 4.4 mg. succinic acid. The CH<sub>2</sub>Cl<sub>2</sub> sol. fraction evapd. gave 37 mg. VIIIa. IX (37.5 mg.) heated 4 hrs. under N with 4 ml. N KOH, the cooled mixt. acidified, and the solid **recrystd.** gave 10 mg. 5-methylocta-2,4-dienedioic acid (XI), feathery needles, m. 195-6.5.degree. (EtCOMe), .lambda. 271 m.mu., .epsilon. 26,780, .nu. 1695, 1685, 1630, and 1610 cm.<sup>-1</sup> Extn. of the aq. filtrate with EtOAc gave 16 mg. product, m. 120-50.degree., but attempts to purify it further were unsuccessful. Ultraviolet absorption measurements indicated the presence of 60% XI.

CC 10B (Organic Chemistry: Aliphatic Compounds)

L66 ANSWER 20 OF 28 HCA COPYRIGHT 2003 ACS on STN

54:28749 Original Reference No. 54:5657d-i,5658a-f Phenoxazine. I. Oxidation products of phenoxazine. Musso, Hans (Univ. Gottingen, Germany). Chemische Berichte, 92, 2862-73 (Unavailable) 1959. CODEN: CHBEAM. ISSN: 0009-2940.

GI For diagram(s), see printed CA Issue.

AB The oxidn. of phenoxazine (I) with a no. of oxidants was investigated and found to give the red-violet phenoxazinyl radical cation which either polymerized or was converted to 2-phenoxazone (II). The polyphenoxazine (III) appeared to have the structure IIIa (R = 3 phenoxazinyl, R' = N-phenoxazinyl) on the basis of a comparison of the spectral properties of 4,9'- (IV) and 2,9'-biphenoxazine (V) with those of III. I (0.92 g.) in 50 cc. Me<sub>2</sub>CO and 20 cc. H<sub>2</sub>O buffered with 1 g. K<sub>2</sub>HPO<sub>4</sub>, treated portionwise at room temp. with 2.80 g. ON(SO<sub>3</sub>K)<sub>2</sub> (VI) in H<sub>2</sub>O, shaken 0.5 hr., extd. with C<sub>6</sub>H<sub>6</sub>, the ext: evapd. in vacuo, and the tacky residue chromatographed from C<sub>6</sub>H<sub>6</sub> on Al<sub>2</sub>O<sub>3</sub> yielded a dark product which dissolved in a little warm dioxane, cooled, and dild. slowly with MeOH gave 445 mg. III, decomp. 240-60.degree. to a dark melt (solns. in org. solvents turn greenish in light); further elution of a red-brown zone gave a brown solid which rechromatographed on silica gel yielded 190 mg. II, red-brown **crystals**, m. 216.5.degree. (sublimed in vacuo). The top portion of the column **scraped** out and eluted with MeOH gave a red-fluorescing soln. of 7-hydroxy-2-phenoxazine (VII). I (183 mg.) in 30 cc. glacial AcOH oxidized with 600 mg. VI in 40 cc. 0.2M NaH<sub>2</sub>PO<sub>4</sub> gave 67% II; the C<sub>6</sub>H<sub>6</sub>-extd. mixt. extd. with BuOH, the ext. evapd. in vacuo, and the residue chromatographed on cellulose with 0.2M phosphate buffer-BuOH (pH 12.25) yielded 14 mg. VII, which acetylated with 3 cc. 1:1 Ac<sub>2</sub>O-pyridine, evapd. in vacuo, and chromatographed from CHCl<sub>3</sub>-EtOAc on silica gel gave 2.7 mg. acetate of VII, orange rodlets, m. 223-6.degree. (decompn.) (sublimed in vacuo). I (157 mg.) in 10 cc. pure Me<sub>2</sub>CO consumed at 0.degree. 1.70 equivs. 0.32N aq. KMnO<sub>4</sub>. I (1.70 g.) oxidized with 2 equivs. KMnO<sub>4</sub>, filtered, evapd., extd. with C<sub>6</sub>H<sub>6</sub>, the ext. evapd. in vacuo, the dark, brittle residue (1.54 g.) chromatographed twice, and repptd. from dioxane with MeOH yielded 1.00 g. III, light gray powder, darkens at 300-30.degree. without melting, mol. wt. 1200. I (3.66

g.) in 100 cc. C<sub>6</sub>H<sub>6</sub> treated dropwise at 5-10.degree. 45 min. with 3.2 g. Br in 20 cc. C<sub>6</sub>H<sub>6</sub>, the mixt. filtered after 1 hr. from 5.90 g. violet-black ppt., the light violet filtrate washed and evapd. in vacuo, and the residue **crystd.** 3 times from C<sub>6</sub>H<sub>6</sub> gave 70 mg. dibromophenoxazine (VIII), light green **crystals**, m. 165-70.degree. (decompn.); filter residue (0.875 g.) in 10 cc. glacial AcOH poured into 200 cc. boiling H<sub>2</sub>O, filtered after 24 hrs. from 0.574 g. ppt., extd. with C<sub>6</sub>H<sub>6</sub>, the ext. chromatographed on Al<sub>2</sub>O<sub>3</sub>, and the crude product sublimed in vacuo gave 2.3 mg. II; the sublimation residue fractionated from C<sub>6</sub>H<sub>6</sub> gave 254 mg. VIII; the mother liquor from the VIII evapd. and **recrystd.** from cyclohexane yielded 120 mg. bromophenoxazine (IX), leaflets, m. 128-35.degree., and 5 mg. I. The crude IX **recrystd.** repeatedly from C<sub>6</sub>H<sub>6</sub>-cyclohexane gave leaflets, m. 135-7.degree. (decompn.), which turned pink within 2 weeks, m. 133.degree. (decompn.). The crude VIII **recrystd.** from glacial AcOH and C<sub>6</sub>H<sub>6</sub> gave needles, m. 165-72.degree.. VIII (300 mg.) refluxed 3 hrs. in 6 cc. Ac<sub>2</sub>O, evapd. in vacuo, and **recrystd.** twice from cyclohexane gave 207 mg. N-Ac deriv. (X) of VIII, needles, m. 224-5.degree. (sublimed in vacuo at 120.degree.). X (4.5 g.) in 250 cc. glacial AcOH treated at 15.degree. under N while being irradiated with a Hg-vapor lamp with 3.2 g. Br in 50 cc. glacial AcOH in portions during 1 hr. and filtered, the residue (3.1 g.) treated with C<sub>6</sub>H<sub>6</sub> and dil. aq. Na<sub>2</sub>CO<sub>3</sub>, the C<sub>6</sub>H<sub>6</sub> phase filtered and evapd., and the residue (1.3 g.) **recrystd.** from C<sub>6</sub>H<sub>6</sub> yielded 150 mg. tetrabromophenoxazine (XI), pale, yellow needles, m. 248-51.degree. (decompn.); the mother liquor gave an addnl. 5 mg. XI and 540 mg. mixt. of IX and a little I; the original filter residue fractionally **crystd.** from C<sub>6</sub>H<sub>6</sub> gave 200 mg. XI. Crude XI **recrystd.** from C<sub>6</sub>H<sub>6</sub> gave needles, m. 250-1.degree.. In all bromination runs, a small amt. of green, basic dyes were also obtained. The mother liquors from the **cryst.** bromination products turned always brown to green and contained considerable amts. of mixed brominated III which were pptd. from C<sub>6</sub>H<sub>6</sub> or dioxane with MeOH as gray-brown or greenish powders. The following colors were observed in concd. H<sub>2</sub>SO<sub>4</sub>: IX blue-violet, VIII blue, XI green-blue. A series of oxidn. runs of I with VI were performed in aq. Me<sub>2</sub>CO at various pH (pH, buffer used, and % yields of III, II, and VII given): 12.6, K<sub>3</sub>PO<sub>4</sub>, 45, less than 1, -; 7.8, K<sub>2</sub>HPO<sub>4</sub>, 48, 25, less than 1; 4.5, KH<sub>2</sub>PO<sub>4</sub>, 5, 63, 1; -, glacial AcOH, less than 1, 62, 6. The color reactions of I with various oxidizing agents were detd. (oxidizing agent and color given): Pb(OAc)<sub>4</sub> in AcOH, red-violet to blue-black to yellow; Br in AcOH and C<sub>6</sub>H<sub>6</sub>, red-violet to blue-violet to light blue; FeCl<sub>3</sub> in AcOH, red-violet (stable); H<sub>2</sub>O<sub>2</sub> in AcOH, red-violet (slowly); air in AcOH, weakly red-violet (slowly); concd. H<sub>2</sub>SO<sub>4</sub>, red-violet (slowly); KMnO<sub>4</sub> in Me<sub>2</sub>CO, light yellow (light red to brown turbidity); VI in AcOH, orange to yellow-orange; VI in aq. Me<sub>2</sub>CO (pH 4.5), orange-red to yellow; VI in Me<sub>2</sub>CO (pH 7.8), light yellow (pinkish white turbidity); VI in Me<sub>2</sub>CO (pH 12.6), red-brown to dark brown.

CC 10G (Organic Chemistry: Heterocyclic Compounds)

L66 ANSWER 21 OF 28 HCA COPYRIGHT 2003 ACS on STN

54:11196 Original Reference No. 54:2243f-g Preparation of monoperphthalic acid. Linholter, S.; Sorensen, P. (Tech. Univ., Copenhagen). Acta Chemica Scandinavica, 12, 1331 (English) 1958. CODEN: ACHSE7. ISSN: 0904-213X.

AB By keeping the reaction mixt. always below -5.degree., the yield (detd. iodometrically) of monoperphthalic acid in alk. H2O2 oxidn was increased from 40-50% to 90-95%. By constantly scraping the crystals from the walls of the reaction flask, foaming was prevented and no phthalic acid resulted.

CC 10E (Organic Chemistry: Benzene Derivatives)

L66 ANSWER 22 OF 28 HCA COPYRIGHT 2003 ACS on STN

52:103809 Original Reference No. 52:18206h-i,18207a-i Syntheses by free-radical reactions. II. Additive dimerizations effected by hydroxyl radicals. Coffman, D. D.; Jenner, E. L. (E. I. du Pont de Nemours & Co., Wilmington, DE). Journal of the American Chemical Society, 80, 2872-7 (Unavailable) 1958. CODEN: JACSAT. ISSN: 0002-7863.

AB A new 1-step synthesis yielding long-chain, unsatd. dicarboxylic acids, diketones, glycols, and other difunctional compds. has been found in the reaction of a 1,3-diene, a monofunctional compd., and hydroxyl radicals generated from H2O2 with FeSO4. (CH2:CH)2 (I) (158 cc.), 34 g. H2O2 in 118cc. H2O, and 278 g. FeSO4.7H2O in 575 cc. H2O and 55 cc. concd. H2SO4 added simultaneously and equivalently at 5.degree. with stirring during 12 min. to 750 cc. Me3COH and 27 cc. H2SO4 in 1500 cc. H2O, the aq. layer extd. with Et2O, and the combined org. layer and ext. worked up yielded 81 g. unsatd. C16-glycol; a sample of the crude glycol fractionated gave a main fraction of [Me2C(OH)CH2CH2CH:CHCH2]2, b0.08 80.degree., nD25 1.4773; the crude glycol mixt. hydrogenated in dioxane at 30.degree. over Pd-C gave [Me2C(OH)(CH2)5]2, m. 50-2.degree. (Et2O). CH2:C(Cl)CH:CH2, H2O2, FeSO4, and H2SO4 (1 mole each) added in the usual manner to 27 cc. H2SO4 and 750 cc. Me3COH in 1500 cc. H2O at 25.degree., the mixt. extd. with Et2O, and the ext. treated with 0.1 g. phenothiazine and distd. up to 80.degree./1 mm. yielded 76% unsatd. dichloroglycol C16H28Cl2O2 as a residue. Isoprene, cyclopentadiene, and CH2:CHCN yielded similarly with Me3COH 46, 20, and 54% additive dimers, resp. I (2 moles) and 1 mole each of H2O2 and FeSO4 added to 27 cc. H2SO4 and 500 cc. H2O in 2000 cc. EtOH, and the product isolated with Et2O yielded 13.2 g. unsatd. glycol which hydrogenated over Pd-C gave a material, b0.8 136.degree., consisting mainly of HO (CH2)12OH. I (2 moles) and 1 mole each of H2O2, FeSO4, and H2SO4 added to 27 cc. H2SO4 and 250 cc. cyclopentanone in 2200 cc. H2O, the mixt. extd. with Et2O, and the ext. worked up yielded 70% unsatd. diketone which hydrogenated at room temp. and 2000 lb. over Pd-C yielded the satd. ketone C18H30O2, b0.25 138-42.degree.. The product from the additive dimerization of I with AcOH was a viscous amber material, sol. in alkali; a portion dissolved in aq. alkali, hydrogenated at 60.degree. and 2000 lb. over Pd-C, acidified with H2SO4, and filtered, the residue refluxed with EtOH and a trace of H2SO4, the

mixt. neutralized with KOAc, the EtOH distd., the crude residue dissolved in PhMe and filtered, and the filtrate distd. yielded the Et ester of a 10-C satd. hydroxy acid, b0.3 150-80.degree..  $\text{CH}_2(\text{CH}_2\text{CO}_2\text{H})_2$  (880 g.), 27 cc.  $\text{H}_2\text{SO}_4$ , and 1500 cc.  $\text{H}_2\text{O}$  treated simultaneously with 2 moles I and 1 mole  $\text{H}_2\text{O}_2$  and a soln. contg. 1 mole each of  $\text{FeSO}_4$  and  $\text{H}_2\text{SO}_4$ , the mixt. extd. with  $\text{Et}_2\text{O}$ , the ext. concd. to about 400 cc. and subjected to a **countercurrent** distribution between 600 cc. each of  $\text{Et}_2\text{O}$  and  $\text{H}_2\text{O}$  gave from the org. phase 58.2 g. C18-tetracarboxylic acid (contg. 2 double bonds). Additive dimerization of I with 750 cc. AcOMe, 27 cc.  $\text{H}_2\text{SO}_4$ , and 1800 cc.  $\text{H}_2\text{O}$  gave in the usual fashion 34.2 g. additive dimer; a 15.2-g. sample heated on the steam bath with 80 cc.  $(\text{CH}_2\text{OH})_2$  and 10 cc. 20M NaOH and evapd. at 105.degree./5 mm., and the residue dild. with  $\text{H}_2\text{O}$  and extd. with  $\text{Et}_2\text{O}$  yielded 2.4 g. C10-glycol, b1 115-30.degree.; the aq. phase acidified with  $\text{H}_2\text{SO}_4$  and extd. with  $\text{Et}_2\text{O}$ ; the ext. neutralized with 75 cc. N NaOH, the aq. soln. of the Na salt acidified with excess  $\text{H}_2\text{SO}_4$ , and the volatile acid distd. gave 42 meq. AcOH and left 12 meq. nonvolatile acid. Additive dimerization of I with  $\text{HCO}_2\text{Me}$  gave 27% dimer which sapond. yielded 25% volatile and 75% nonvolatile acids. Additive dimerization of I with AcH yielded 7% dimer contg. both CHO and Ac groups. Maleic acid,  $\text{H}_2\text{O}_2$  and  $\text{FeSO}_4$  (1 mole each) dimerized in a large excess  $\text{Me}_3\text{COH}$ , the mixt. extd. with  $\text{EtCOMe}$ , and the ext. evapd. yielded 53 g. crude dihydroxytetracarboxylic acid which **recrystd.** from  $\text{H}_2\text{O}$  yielded the dilactone, m. 180-220.degree. (decompn.). The lactonic acid treated with  $\text{CH}_2\text{N}_2$  yielded the di-Me ester, m. 173-5.degree..  $\text{Me}_2\text{CO}$  (600 cc.) and 150 cc.  $\text{H}_2\text{O}$  treated during 20 min. at -5.degree. with 125 cc. 30%  $\text{H}_2\text{O}_2$ , 189 cc. I, and 1200 cc. 1M  $\text{FeSO}_4$  (1M in  $\text{H}_2\text{SO}_4$ ), the aq. layer extd. with  $\text{Et}_2\text{O}$ , and the combined org. layer and ext. neutralized with  $\text{NaHCO}_3$ , dried, and distd. gave the following fractions (wt. in g., b.p./mm.,  $n_{\text{D}}^{25}$ , and characterization given): (1) 7.5, 33-44.degree./26, 1.3921, mixt. of AcOH and hydrocarbons; (2) 3.8, 47-61.degree./26, 1.4291, chiefly hydrocarbon; (3) 6.1, 63-7.degree./26, 1.4321, probably 3-ethyl-1,5-octadiene; (4) 8.5, 44-6.degree./4.5, 1.4351, 3,7-decadiene (II); (5) 4.2, 46.degree./4.5, 1.4365, II; (6) 3.6, 78-89.degree./4.5, 1.4551, hydrogenation equiv. 72 (the hydrogenated material gave a noncryst. 3,5-dinitrobenzoate); (7) 4.4, 88-9.degree./4.5, 1.4589, mixt. of ketone and alc.; (8) 3.3, 74-9.degree./1.5, 1.4571, -; (9) 4.3, 80-2.degree./1.5, 1.4549, hydrogenation equiv. 88, carbonyl equiv. 214; (10) 4.7, 87-9.degree./1.5, 1.4561, hydrogenation equiv. 95, carbonyl equiv. 204; (11) 3.3, 88-101.degree./0.3, 1.4630, -; (12) 2.9, 100-11.degree./0.3, 1.4742, mixt. of glycol, diketone, and hydroxy ketone; (13) 1.0, 110-20.degree./0.3, 1.4735, -; and 3.0 g. residue. II (1.8 g.) treated with Br in  $\text{CCl}_4$  gave 3,4,7,8-tetrabromodecane, m. 68-9.degree. ( $\text{CCl}_4$ ). The hydrogenated product from fraction 10 gave a 2,4-dinitrophenylhydrazone, m. 78-9.degree.. The solubilities of I at 25.degree. and I atm. (given in vol. gas/vol. solvent) were detd. for the following solvents:  $\text{H}_2\text{O}$  0.3; 33% aq. AcOH 0.87; 33% aq.  $\text{EtCO}_2\text{H}$  1.4; 33% aq.  $\text{Me}_3\text{COH}$  3.5. Additive dimerization of I with BuOH, iso-PrOH, and  $\text{EtCO}_2\text{H}$  yielded 47, 13,

and 77% product, resp.

CC 10B (Organic Chemistry: Aliphatic Compounds)

L66 ANSWER 23 OF 28 HCA COPYRIGHT 2003 ACS on STN

50:12037 Original Reference No. 50:2446a-i,2447a-d Cycloalkanone peroxides. I. Preparation of "cyclohexanone peroxide" by oxidation of cyclohexanol. Brown, Northrop; Hartig, Martval J.; Roedel, Milton J.; Anderson, Arthur W.; Schweitzer, Carl E. (E. I. du Pont de Nemours & Co., Inc., Wilmington, DE). Journal of the American Chemical Society, 77, 1756-9 (Unavailable) 1955. CODEN: JACSAT. ISSN: 0002-7863.

AB The autocatalyzed liquid phase oxidation of cyclohexanol (I) with O produced in high yields at moderate conversions a mixt. of peroxides which were qualitatively equiv. to those produced by the reaction of cyclohexanone with H<sub>2</sub>O<sub>2</sub>. Reactions of these peroxides with ferrous ion in hydrocarbon soln. gave 68% HO<sub>2</sub>C(CH<sub>2</sub>)<sub>10</sub>CO<sub>2</sub>H (II). Evidence is presented concerning the structure of these peroxides and the equil. between the various forms. I (280 g.) and 2.8 g. com. cyclohexanone peroxide (III) m. 47-58.degree. to 72-4.degree., treated with stirring at 350 r.p.m. with a stream of 300 cc. O/min., the mixt. heated rapidly to 125.degree., and the oxidation terminated after about 10 l. O had been consumed gave a mixt. contg. about 9-10 mole-% peroxide; in oxidation runs beyond this point, the reaction temp. was reduced progressively; at the end of the run, the mixt. was cooled to room temp. I (2.771 moles), 0.023 mole peroxidic O, and 1.4 g. CaCO<sub>3</sub> treated 2.5 hrs. at 125.degree. with 0.398 mole O, the mixt. added with stirring at 25.degree. during 1 hr. to 48.9 g. Na<sub>2</sub>SO<sub>3</sub> in 489 cc. H<sub>2</sub>O, the mixt. treated with stirring with 9.7 g. Na<sub>2</sub>SO<sub>3</sub>, 97 cc. H<sub>2</sub>O, and 37 cc. AcOH, stirred 1.5 hrs., neutralized to phenolphthalein with 50% aq. NaOH, and steam distd., and the distillate dried and fractionated gave 2.327 moles I and 0.387 mole cyclohexanone (IV). Com. III (10.0 g.) in 100 cc. MeOH treated as rapidly as possible with 60 cc. H<sub>2</sub>O, the mixt. filtered immediately, and the residue dried 1 hr. at room temp. at 1 mm. gave 6.3 g. 1-hydroxy-1'-hydroperoxydicyclohexyl peroxide (V), m. 73.degree.; the mother liquor worked up gave an ill-defined **cryst**. peroxide, m. 38-58.degree., with a peroxide equiv. wt. of 66.8. IV (1 mole) treated with 2 moles anhyd. H<sub>2</sub>O<sub>2</sub>, and the resulting sirup kept several hrs. at 0.degree. and **crystd.** from Et<sub>2</sub>O at -40.degree. gave 1,1'-dihydroxydicyclohexyl peroxide (VI), m. 68.degree.. Deaerated MeOH (700 cc.) added under N to 72.3 g. solid FeSO<sub>4</sub>.7H<sub>2</sub>O, the soln. treated immediately with 24.6 g. com. III in 300 cc. deaerated MeOH at room temp. with stirring during 2-5 min. under N, the mixt. brought to pH 11 with 2N NaOH, the MeOH removed on a steam bath, the residual mixt. boiled 1 hr., cooled, and filtered, the filter cake slurried twice with H<sub>2</sub>O contg. a little NaOH, the combined filtrate and washes acidified with concd. H<sub>2</sub>SO<sub>4</sub>, boiled to remove MeOH and volatile acids, and cooled, and the solid deposit filtered and dried at 80.degree. gave 12.3 g. crude II, m. 125-6.5.degree. (**recrystd.** twice from C<sub>6</sub>H<sub>6</sub>, m. 127.5.degree.); the yields of II from III ranged from 48 to 62%. A similar run in MeOH after 18 hrs. gave 18% II. V gave in the same



manner 78% II and VI gave 23%. The peroxides were sepd. from the crude I oxidation product (9.5% conversion to peroxides) by a **countercurrent** distribution with mutually satd. I and H<sub>2</sub>O in 16 transfers. In soln., com. III undergoes slow dissocn. to an equil. contg. monomeric **forms** and possibly free H<sub>2</sub>O<sub>2</sub> as well. A qual. measurement of the degree of dissocn. could be obtained by a single-stage solvent partition, the extent of dissocn. measured by the relative soly. in the aq. and org. phases. To measure the dissocn. of III in various solvents, 10% solns. were kept at room temp., and 1-cc. samples equilibrated after certain time intervals between 20 cc. each of xylene and H<sub>2</sub>O-12% MeOH, and the layers sepd. and analyzed for peroxide; the solvents used and the % total peroxide in the aq. phase after 5-10 min., 1.5, 5, and 18 hrs. were: MeOH, 48.3, 67.2, -, 74.3; MeOH-0.1% H<sub>2</sub>SO<sub>4</sub>, 60.3, -, -, 61.6; MeOH-0.1% NaOH, 82.5, -, -, 81.5; AcOH, 73.6, -, 74.3, -; MeCN (5%), -, 67.7 (45 min.), 73.8 (2.7 hrs.), -; MeNO<sub>2</sub> (5%), 61.3, 62.0, -, -; Me<sub>2</sub>CO, 45.8, 59.8, -, 75.2; CHCl<sub>3</sub>, 45.5, -, 71.2, 71.0 (addn. of 16% I to this soln. caused a decrease to 55% H<sub>2</sub>O-sol. peroxides in 30 min.); C<sub>6</sub>H<sub>6</sub> (8%), 48.4, 63.8, -, 61.5; Me<sub>3</sub>COH, 46.1 (15 min.), -, 61.7, -; dioxane, 43.2, 44.6, -, -; Et<sub>2</sub>O, 41.1, 43.8, -, -. The data show that the dissocn. to H<sub>2</sub>O sol. peroxides occurred most rapidly and completely in solvents of high dielec. consts.; in the presence of acids and bases the dissocn. was extremely rapid. I (224.65 g.) and 2.46 g. com. III oxidized by the standard method, the product treated with 179.49 g. IV, the mixt. kept at room temp. overnight, blown with N, added with stirring at room temp. to 930 cc. ferrous heptanoate in C<sub>6</sub>H<sub>6</sub> contg. 0.338 equiv. ferrous ion, dild. with H<sub>2</sub>O, neutralized with NaOH, and distd., the residue digested 2 hrs. with excess NaOH, filtered, washed with Et<sub>2</sub>O, acidified with H<sub>2</sub>SO<sub>4</sub>, and steam distd., and the distn. residue **recrystd.** gave 19.30 g. crude acid; the raffinate evapd. to dryness and extd. with Et<sub>2</sub>O and CHCl<sub>3</sub> yielded 4.89 g. crude II, dark semicryst. material which decolorized with C and **recrystd.** from C<sub>6</sub>H<sub>6</sub> and aq. MeOH gave pure II, m. 127.degree.. The steam distillate fractionated gave 190.97 g. unchanged I and 185.24 g. IV. Caproic acid (69.6 g.) and 100 cc. distd. H<sub>2</sub>O neutralized to phenolphthalein with 2N NaOH, the mixt. treated with 34.8 g. addnl. caproic acid, treated at room temp. under N with 550 cc. O-free C<sub>6</sub>H<sub>6</sub> with stirring and then during about 15 min. with 83.4 g. FeSO<sub>4</sub>.7H<sub>2</sub>O in 600 cc. H<sub>2</sub>O, the upper layer washed 3 times with 400-cc. portions O-free H<sub>2</sub>O and treated with 5 g. Fe powder, and the residual H<sub>2</sub>O distd. off at atm. pressure gave the ferrous soap reagent which was stored and handled under N. The following % yields of II (given) were obtained from I by reductive coupling of the resulting peroxides: 44 with FeSO<sub>4</sub>-MeOH, 45 with Fe(OAc)<sub>2</sub>-50% dioxane, 64 with ferrous caproate-PhMe. Com. III gave 47% II with FeSO<sub>4</sub>-MeOH, and 65% with ferrous caproate-PhMe.

CC 10 (Organic Chemistry)

L66 ANSWER 24 OF 28 HCA COPYRIGHT 2003 ACS on STN

49:23810 Original Reference No. 49:4597b-h Halodiphenacyls. I.

Stevens, Calvin L.; Church, Robert J.; Traynelis, Vincent J. (Wayne

Univ., Detroit, MI). Journal of Organic Chemistry, 19, 522-32 (Unavailable) 1954. CODEN: JOCEAH. ISSN: 0022-3263.

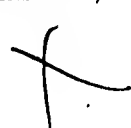
AB The structures of .alpha.- and .beta.-chloro-, -bromo-, and iodo-diphenacyls, resp., have been correlated by phys. (ultraviolet and infrared absorption spectra, which are given) and chem. means. Adding (20 min.) 6.2 g. KOH in 75 cc. MeOH to 19.9 g. BzCH<sub>2</sub>Br at 0-5.degree. and stirring the mixt. 45 min. at 5.degree. give 10.5 g. of a diphenacyl mixt. which is sepd. by fractional **crystn.** into 16% .alpha.-bromodiphenacyl (I), m. 135-6.degree., and 34% .beta.-bromodiphenacyl (II), m. 160-1.degree.. Similarly, 15.5 g. BzCH<sub>2</sub>Cl gives 20% .alpha.-chlorodiphenacyl (III), m. 116-17.degree. and 47% .beta.-isomer (IV), m. 147-8.degree.. Refluxing 320 mg. I and 300 mg. NaI in Me<sub>2</sub>CO gives 86% .alpha.-iododiphenacyl (V), decomp. 71-2.degree.. V is unstable and decomp. on standing. Similarly 550 mg. III and NaI give only 20 mg. V. Refluxing 950 mg. II and 680 mg. NaI in Me<sub>2</sub>CO gives 80% .beta.-iododiphenacyl (VI), decomp. 110-10.5.degree.. When 550 mg. IV and NaI are refluxed 24 hrs. 22% VI is obtained. Treating 500 mg. II in 50% ether-C<sub>6</sub>H<sub>6</sub> with 25 cc. ether contg. 500 mg. HBr 24 hrs. in the cold, concg. the soln., and dilg. it with petr. ether give 56% II-HBr adduct (VII), m. 144-5.degree. (decompn.). Keeping 1 g. VII 1 hr. in 100 cc. liquid NH<sub>3</sub> and evapg. the NH<sub>3</sub> give 69% I. Refluxing gently 11.1 g. dypnone, 8.9 g. (CH<sub>2</sub>CO)<sub>2</sub>NBr, and 0.3 g. Bz<sub>2</sub>O<sub>2</sub> in 60 cc. anhyd. CCl<sub>4</sub> 0.5 hr., adding another 0.3 g. Bz<sub>2</sub>O<sub>2</sub>, refluxing another hr., and evapg. the filtered soln. give 51% bromodypnone (.beta.-bromomethylchalcone) (VIII), light yellow needles, m. 66-7.degree.. Adding at 0.degree. 2.7 g. SO<sub>2</sub>Cl<sub>2</sub> to 4.4 g. dypnone and refluxing the mixt. gently 23 hrs. give 42% .beta.-chloromethylchalcone (IX); pale yellow needles, m. 74-5.degree.. Treating 3 g. VIII in 25 cc. anhyd. Me<sub>2</sub>CO 1 hr. at 20.degree. with 1.5 g. NaI and pouring the filtered soln. into 50 cc. ice H<sub>2</sub>O give 90% .beta.-iodomethylchalcone (X), pale yellow platelets, m. 74-5.degree. (decompn.). Similarly, 0.8 g. IX and NaI in Me<sub>2</sub>CO 1.5 hrs. at 20.degree. give 71% X. Adding 1 cc. N NaOH and 5 cc. 15% H<sub>2</sub>O<sub>2</sub> to 1.2 g. VIII in 90 cc. MeOH at 0.degree. keeping the mixt. 0.5 hr., adding 5 cc. 15% H<sub>2</sub>O<sub>2</sub> to the filtered soln., keeping the mixt. 1.5 hrs. in the refrigerator, and fractionally **crystg.** the ppt. (83%) from abs. EtOH give 49% I and 12% II. Treating similarly 0.51 g. IX with H<sub>2</sub>O<sub>2</sub> gives 51% III and 16.5% IV, and 0.35 g. X and H<sub>2</sub>O<sub>2</sub> give 39% V and 8% VI. When dypnone is treated with H<sub>2</sub>O<sub>2</sub> 64% low-melting dypnone oxide, m. 93-4.degree., and 7% high-melting isomer, m. 160-1.degree., are obtained. The structures of the halodypnones are proved by the spectral studies and from these data the low-melting .alpha.-halodiphenacyls are considered to have the trans-configuration.

CC 10 (Organic Chemistry)

L66 ANSWER 25 OF 28 HCA COPYRIGHT 2003 ACS on STN

48:31501 Original Reference No. 48:5633i,5634a-b **Hydrogen**

**peroxide:** the low-temperature heat capacity of the solid and the third-law entropy. Giguere, Paul A.; Liu, I. D.; Dugdale, J.



S.; Morrison, J. A. (Laval Univ., QC). Canadian Journal of Chemistry, 32, 117-28 (Unavailable) 1954. CODEN: CJCHAG. ISSN: 0008-4042.

- AB The heat capacity of **cryst. H<sub>2</sub>O<sub>2</sub>**, 99.97% pure, between 12.degree.K. and the m.p., was detd. with a low-temp. adiabatic calorimeter. The heat of fusion is 2987  $\pm$  3 cal./mole. The triple point was estd. to be 272.74.degree.K. The only anomaly in the heat-capacity measurements was the absorption of 1.3 cal./mole at 216.8  $\pm$  0.15.degree.K., the lower eutectic temp. of H<sub>2</sub>O-H<sub>2</sub>O<sub>2</sub> solns. The entropy of **H<sub>2</sub>O<sub>2</sub>** as an ideal gas at 1 atm. and 25.degree. is 55.76  $\pm$  0.12 cal./mole degree. Comparison of this datum with the recalcd. statistical entropy leads to a value of 3.5 kcal./mole for the height of a hypothetical single barrier hindering internal rotation in the mol. It is concluded that **H<sub>2</sub>O<sub>2</sub>** does not consist of two tautomeric modifications.
- CC 2 (General and Physical Chemistry)
- IT Heat of **fusion**  
Triple point  
(of **hydrogen peroxide**)
- IT Entropy  
Heat capacity  
(of **hydrogen peroxide** (solid))
- IT Molecules  
(rotation of, of **H<sub>2</sub>O<sub>2</sub>**, energy barrier to)
- IT Energy  
(to rotation in hydrocarbons, for rotation in **H<sub>2</sub>O<sub>2</sub>**)
- IT **7722-84-1, Hydrogen peroxide**  
(entropy and heat capacity of solid)

- L66 ANSWER 26 OF 28 HCA COPYRIGHT 2003 ACS on STN 44:19556 Original Reference No. 44:3828f-i,3829a-f The reaction between the isopolymolybdates and hydrogen peroxide. I.
- Martinez, J. Beltran (Univ. La Laguna, Spain). Anales real soc. espan. fis. y quim. (Madrid), 45B, 665-96 (Unavailable) 1949.
- AB The addn. of **H<sub>2</sub>O<sub>2</sub>** to NH<sub>4</sub> bi-, tri-, para-, and tetra-molybdates (H<sub>2</sub>O<sub>2</sub>: MoO<sub>3</sub> = 2 or more mols.: 1 mol.) gives rise to the same peroxide compd., diammonium tetraperoxydimolybdate (NH<sub>4</sub>)<sub>2</sub>O.4O.2MoO<sub>3</sub>.xH<sub>2</sub>O (I) in each case. In the analyses below, active-oxygen (OA) estn. gave variable results with acid KMnO<sub>4</sub>, probably because the H<sub>2</sub>SO<sub>4</sub> liberated permolybdic acids. The peroxy compd. was, therefore, dissolved in excess standard neutral KMnO<sub>4</sub>, acidified at once with H<sub>2</sub>SO<sub>4</sub>, and the excess KMnO<sub>4</sub> estd. by adding excess FeSO<sub>4</sub> and titrating with the same KMnO<sub>4</sub> soln. The MoO<sub>3</sub> was estd. gravimetrically; the NH<sub>4</sub>, by addn. of NaOH and distn. To 10-ml. portions of diammonium dimolybdate (constitution checked by cond. measurement) contg. 3.17 .times. 10-3 mols. MoO<sub>3</sub> and dild. to 100 ml., increasing amts. of 8.92 M **H<sub>2</sub>O<sub>2</sub>** were added, and the solns. were analyzed conductometrically and also potentiometrically with 0.102 N NaOH and a calomel half-cell. This compd. behaves in soln. as a mixt. of (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> and H<sub>2</sub>MoO<sub>4</sub>. With no **H<sub>2</sub>O<sub>2</sub>** present 3.162 .times.

10-3 mols. NaOH neutralized the "free" acid. Increasing amts. of **H2O2** shifted the potential jump corresponding to neutralization of "free" acid until it became tangent to the ordinate axis at 0.7 ml. (or more) of 8.92 M **H2O2** added. All the "free" acid was removed from soln. by 6.244 .times. 10-3 mols. **H2O2**, i.e. 2 mols. **H2O2** react with each mol. MoO3. The reaction is probably  $(\text{NH}_4)_2\text{O} \cdot 2\text{MoO}_3 + 4\text{H}_2\text{O}_2 \rightarrow \text{I} + 4\text{H}_2\text{O}$ . I, which behaves as a neutral salt in presence of NaOH. I was prepd. by adding 10 g. ammonium paramolybdate to 38 ml. 0.2475 N  $\text{NH}_4\text{OH}$ , adding 25 ml. 8.9 M **H2O2**, and pptg. with alc. Analysis: 14.4% OA; 44.58% Mo. Diammonium diperoxydimolybdate with OA:Mo = 1:1 was prepd. by dissolving 10 g. ammonium paramolybdate in 7.5 ml. 9 M **H2O2** and adding 38 ml. 0.2475 N  $\text{NH}_4\text{OH}$ . The color changed from orange-yellow to pale yellow. The soln. was cooled on ice and pptd. with alc. Analysis: 6.93% OA; 48.29% Mo; 8.99%  $\text{NH}_3$ , corresponding to  $(\text{NH}_4)_2\text{O} \cdot 2\text{OA} \cdot 2\text{MoO}_3 \cdot 2\text{H}_2\text{O}$ . Ammonium trimolybdate,  $(\text{NH}_4)_2\text{O} \cdot 3\text{MoO}_3$ , was confirmed by cond. and potentiometric measurements. Cond. analysis showed **H2O2** added 0.5 **H2O2** and 2 **H2O2** per Mo. Excess **H2O2** gives I. Diammonium triperoxytrimolybdate was prepd. by dissolving 5 g. ammonium trimolybdate in 10 ml. 9 M **H2O2** and evapg. in a current of air at 45-50.degree.. An orange-yellow compd. formed when the soln. had been evapd. to a sirup. It was cooled and **scraped** and the whole mass **crystd.** to an orange-yellow powder, contg. 48.7% Mo and 9.53% OA, corresponding to  $(\text{NH}_4)_2\text{O} \cdot 3\text{OA} \cdot 3\text{MoO}_3 \cdot x\text{H}_2\text{O}$ . Diammonium tetramolybdate,  $(\text{NH}_4)_2\text{O} \cdot 4\text{MoO}_3 \cdot 2\text{H}_2\text{O}$ , was confirmed by cond. measurement. Cond. analysis showed that **H2O2 forms** a peroxy compd. with 2 OA:1 Mo; previous expts. showed it is a dimolybdate and cond. analysis showed the depolymerization. This compd. was isolated by heating gently 10 g. diammonium tetramolybdate in 25 ml. 8.9 M **H2O2**, cooling on ice, and pptg. with a large excess (300 ml.) of alc. The lemon-yellow ppt. contained 14.75% OA and 45.97% Mo, corresponding to I. Diammonium tetraperoxytetramolybdate was prepd. by dissolving 4 g. diammonium tetramolybdate in 10 ml. 9 M **H2O2** and evapg. in an air current at 45-50.degree. to a sirup. It **crystd.** when cooled. Analysis: 7.7% OA; 48.3% Mo. Potentiometric analysis showed that it is a (peroxy) deriv. of diammonium tetramolybdate. A soln. of  $\text{NH}_4$  paramolybdate contg. 22.2 .times. 10-4 mols. Mo gave a potentiometric jump for neutralization of "free" acid when 25.34 .times. 10-4 mols. NaOH was added. This corresponds very closely to Na:Mo = 8:7, which confirms Delafontaine's formula  $((3(\text{NH}_4)_2\text{O} \cdot 7\text{MoO}_3))$ . Cond. analysis showed that **H2O2** adds in the ratios **H2O2:MoO3** = 2:1 and 1:1. The 1:1 compd. was that prepd. by Muthman and Nagel (Z. anorg. Chem. 17, 73(1898)). To isolate the 2:1 compd. 12 g.  $\text{NH}_4$  paramolybdate in 12 ml. 9 M **H2O2** was pptd. by letting-drop into abs. alc. The yellow-white salt was washed and dried at room temp. Analysis: 15.04% OA, 44.91% Mo; 8.25%  $\text{NH}_3$ , corresponding to I.

CC 6 (Inorganic Chemistry)  
IT Molybdates

- (isopoly-, reactions with H<sub>2</sub>O<sub>2</sub>)
- IT Ammonium peroxydymolybdates  
(formation in reaction of isopolymolybdates with H<sub>2</sub>O<sub>2</sub>)
- IT Ammonium molybdates  
(reaction with H<sub>2</sub>O<sub>2</sub>)
- IT 7722-84-1, **Hydrogen peroxide**  
(reactions of, with isopolymolybdates)

L66 ANSWER 27 OF 28 HCA COPYRIGHT 2003 ACS on STN

12:13426 Original Reference No. 12:2289d-h Double catalytic action in the oxidation of aluminium in the presence of mercury; oxidation of aluminium powder at ordinary temperature. Ronceray, P. Bulletin des Sciences Pharmacologiques, 25(Pt. 2), 193-8 (Unavailable) 1918. CODEN: BSPHAV. ISSN: 0366-3493.

AB Existing theories are reviewed. It is shown that intimate contact with Fe does not render Al oxidizable, but that the very finely divided particles of Al obtained by rubbing the 2 metals together are oxidized by H<sub>2</sub>O, the ease of oxidation depending on the fineness of division. The soln. of Al in Hg results in an extremely fine dispersion of the Al and the particles at the surface of the Hg in contact with air oxidize immediately. In this case the role of the Hg is entirely physical, playing a part solely through its comminuting action on the Al. However, in the case of Al "activated" by Hg the Hg is a true catalyst. In the case of Schoenbein's reaction, formation of a blue color when tincture of guaiac is shaken in the air with Hg (J. prakt. Chem. 1851, 54), the catalyst is not the Hg, but HgO. If pure Hg is used no color is produced, nor does a color **form** on adding H<sub>2</sub>O<sub>2</sub>. Moreover, if an emulsion of tincture of guaiac is shaken with Hg no color is produced, but the Hg becomes very finely divided and is partially oxidized; if the supernatant liquid is then poured off and alc. added, a blue color immediately appears, owing to the insol. resin having been deposited on the finely divided, partially oxidized, Hg. The fact that HgO was actually the catalyst was established by shaking pptd. HgO with emulsified guaiac tincture, when a blue color developed in the presence or absence of H<sub>2</sub>O<sub>2</sub>. HgO also catalyzes the oxidation of Al, as a freshly **scraped** sheet of Al, rubbed with traces of HgO, whitens in the air and decomps. H<sub>2</sub>O. This is possibly a second catalytic function of the Hg in "activated" Al.

CC 6 (Inorganic Chemistry)

L66 ANSWER 28 OF 28 HCA COPYRIGHT 2003 ACS on STN

3:14612 Original Reference No. 3:2679h-i,2680a-b Contributions to the Chemistry of the Cholesterol Group I. The Action of Hydrogen Peroxide and of Fused Potassium Hydroxide on Cholesterol. Pickard, R. H.; Yates, J. Journal of the Chemical Society, Abstracts, 93, 1678 (Unavailable) 1909. CODEN: JCSAAZ. ISSN: 0590-9791.

AB When cholesteryl acetate in AcOH is oxidized with H<sub>2</sub>O<sub>2</sub>, a **cryst.** mixture of the mono- and diacetates of a "triol," C<sub>27</sub>H<sub>45</sub>O<sub>3</sub>, is obtained. These can be separated by extraction of the

diacetate with petroleum ether. The diacetate m. 165.degree. and the monoacetate m. 212.degree.. The "triol" **cryst.** as lustrous needles, m. 239.degree.. The diacetate and dipropionate, m. 172.degree., have been prepared by treating this substance with the corresponding anhydrides. By oxidizing the monoacetate in ACOH sol. with CrO3 and deacetylating with NaOCH3 a keto-alcohol results, C27H44O3, pearly needles, m. 232.degree.. The acetyl derivative obtained above before deacetylation m. 233.degree.. The phenylhydrazone was obtained as orange needles, m. 164.degree.. By oxidizing the "triol" and the keto-alcohol with CrO3, dehydrochlorestenediol, C27H42O3 (oxychlorestenediol), was obtained. By fusing chloresteryl with KOH an acid, C27H46O3, m. 241.degree., can be obtained. It does not decolorize Br or KMnO4. The common salts are gelatinous. Acetyl derivative, m. 163.degree., **cryst.** in prisms. Ethyl ester, C29H50O6, **cryst.** in plates, m. 62.degree.. By treatment of the fusion products with EtOAc and EtNO3 a dibasic acid, C26H44O4, was obtained as long needles, m. 190.degree.. The common salts are amorphous.

CC 10 (Organic Chemistry)

=> d 180 1-6 cbib abs hitind

L80 ANSWER 1 OF 6 HCA COPYRIGHT 2003 ACS on STN

133:210226 Processes of producing a titanium oxide-forming solution and a dispersion with crystalline titanium oxide particles. Ichinose, Hiromichi (Saga Prefecture, Japan). Eur. Pat. Appl. EP 1031538 A1 20000830, 28 pp. DESIGNATED STATES: R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO. (English). CODEN: EPXXDW. APPLICATION: EP 2000-103431 20000225. PRIORITY: JP 1999-50868 19990226; JP 1999-50869 19990226; JP 1999-224190 19990806.

AB The invention provides a process of producing a TiO2-forming soln., wherein a H2O2 soln. is added to a Ti-contg. starting aq. soln. to form a peroxotitanium complex, a basic substance is then added to the peroxotitanium complex to obtain a soln. which is in turn let stand or heated, thereby forming a ppt. of a peroxotitanium hydrate polymer, at least a dissolved component derived from the Ti-contg. starting aq. soln., except water, is then removed from the ppt., and a H2O2 soln. is finally allowed to act on a dissolved component-free ppt. The invention also provides a dispersion contg. TiO2.

IC ICM C01G023-047

ICS C09D017-00

CC 49-3 (Industrial Inorganic Chemicals)

IT Disperse systems

#### Suspensions

(with **cryst.** titanium oxide particles)

IT 7440-32-6, Titanium, processes 7550-45-0, Titanium tetrachloride, processes 7722-84-1, **Hydrogen peroxide**, processes

(in **prodn.** of titanium oxide-forming soln. and

dispersion with cryst. titanium oxide particles)

L80 ANSWER 2 OF 6 HCA COPYRIGHT 2003 ACS on STN

120:11260 Manufacture of calcium peroxide from calcium salts, hydrogen peroxide, and ammonium hydroxide. Savoskina, Anna I.; Salamatina, Inna V.; Zavyalova, Tatyana Z.; Dubrovskij, Arkadij Ya.; Pazdnikov, Igor P. (Vnii reaktivov khim chistykh materialov dlya elektronnoj tekhniki, USSR). U.S.S.R. SU 1756268 A1 19920823 From: Izobreteniya 1992, (31), 82. (Russian). CODEN: URXXAF. APPLICATION: SU 1990-4841682 19900625.

AB To accelerate the process, the reactor is primed with an aq. **suspension** of **cryst. CaO<sub>2</sub>·8H<sub>2</sub>O** before simultaneously feeding a 0.8-2.5M aq. soln. of Ca(NO<sub>3</sub>)<sub>2</sub> or CaCl<sub>2</sub> (as the Ca salt), H<sub>2</sub>O<sub>2</sub>, and NH<sub>4</sub>OH in mol. ratio 1.0:(1.3-1.4):(2.6-2.8). The reaction mixt. is cooled under agitation, and the ppt. is sepd., washed, and thermally dehydrated.

IC ICM C01B015-043

CC 49-3 (Industrial Inorganic Chemicals)

IT 60762-59-6

(priming with, in calcium peroxide manuf. by reacting calcium salts with ammonium hydroxide and **hydrogen peroxide**, for prodn. rate)

L80 ANSWER 3 OF 6 HCA COPYRIGHT 2003 ACS on STN

119:134428 Mechanism of bactericidal effect of helium-neon laser irradiation on Streptococcus mutans (PS 14). Hirano, Yasuyuki; Nishikata, Junichi; Shiomi, Noboru; Kishida, Osamu; Obata, Jun; Ito, Koichi (Sch. Dent., Nihon Univ., Japan). Nippon Shishubyo Gakkai Kaishi, 35(1), 157-61 (Japanese) 1993. CODEN: NSKADI. ISSN: 0385-0110.

AB A study was conducted to examine the mechanism of the bactericidal effect of He-Ne laser irradiation on S. mutans, and to clarify the conditions under which the bactericidal effect was expressed. Two mL of 0.5% agar plus 0.5 mL of bacterial cell **suspension** contg. **crystal violet** (CV) were poured onto brain heart infusion agar, and the surface of the agar plates was vertically irradiated with a He-Ne laser (Acusoft, 10 mW, 632.8 nm, Osada Elec. Co., Tokyo, Japan). After laser irradiation, the plates were cultured anaerobically and also aerobically. Similarly, 0.13 mL of cell suspension contg. CV (final concn.: 3.2 µg/mL) was placed in each well of a microtitration plate, and the laser beam was used to vertically irradiate the cell suspensions. It was found that the bactericidal effect of He-Ne laser irradiation on S. mutans occurred only in the presence of oxygen. Moreover, the cell survival ratio was reduced as the irradiation time was extended. On the other hand, the bactericidal effect was effectively inhibited by superoxide dismutase and catalase. It is concluded that S. mutans cells are quickly killed by He-Ne laser irradiation in the presence of CV and oxygen. Under aerobic conditions, superoxide anions and **hydrogen peroxide** are generated by the photodynamic action of CV, and then S. mutans cells are killed by these bactericidal products.

CC 8-3 (Radiation Biochemistry)  
Section cross-reference(s): 10

L80 ANSWER 4 OF 6 HCA COPYRIGHT 2003 ACS on STN

104:222894 Pipecolic acid is oxidized by renal and hepatic peroxisomes.  
Implications for Zellweger's cerebro-hepato-renal syndrome (CHRS).  
Zaar, Kurt; Angermueller, Sabine; Voelkl, Alfred; Fahimi, H. Dariush  
(Dep. Anat., Univ. Heidelberg, Heidelberg, D-6900, Fed. Rep. Ger.).  
Experimental Cell Research, 164(1), 267-71 (English) 1986. CODEN:  
ECREAL. ISSN: 0014-4827.

AB Increased levels of pipecolic acid have been reported in patients  
with cerebro-hepato-renal syndrome (CHRS) of Zellweger, and the  
general deficiency of peroxisomal function has been implicated in  
its pathogenesis. Therefore, the capacity of normal peroxisomes to  
metabolize pipecolic acid was investigated. Highly purified  
peroxisomes were obtained from rat liver and rat and beef kidney  
cortex. These preps. oxidized D,L-pipecolic acid as evidenced by  
the measurement of **H2O2 prodn.** Incubation with  
either the D- or L-isomer revealed that D-pipecolate is oxidized  
almost exclusively. The specific activities proved to be 20-50  
times higher in renal than in hepatic peroxisomes. A com. available  
**cryst. suspension** of D-amino acid oxidase from  
porcine kidney also oxidized the pipecolic acid with the following.  
rates 54:36:1 resp. for D-:D,L-:L-isomers. Incubation of vibratome  
sections of rat kidney and liver in a medium contg. D-pipecolic acid  
and cerous ions, revealed electron-dense deposits over the matrix of  
peroxisomes. These observations demonstrate the capability of  
mammalian peroxisomes to oxidize pipecolic acid and suggest that the  
absence or deficiency of peroxisomal D-amino acid oxidase may be  
implicated in the pathogenesis of hyperpipecolatemia in Zellweger's  
CHRS.

CC 14-15 (Mammalian Pathological Biochemistry)  
Section cross-reference(s): 13

L80 ANSWER 5 OF 6 HCA COPYRIGHT 2003 ACS on STN

104:112274 Preparation of potassium peroxymonosulfate triple salt.  
Reh, Wolfgang; Schellé, Siegfried (Peroxid-Chemie G.m.b.H., Fed.  
Rep. Ger.). Ger. Offen. DE 3427119 A1 19860123, 10 pp. (German).  
CODEN: GWXXBX. APPLICATION: DE 1984-3427119 19840723.

AB Triple salt 2KHSO5.KHSO4.K2SO4 is prepd. by continuously making an  
aq. working soln. contg. KHSO5, H2SO4, and K2SO4 in a molar ratio of  
(1.3-2.5):(1.2-2.0):1 and concg. in an evapn. app. at low pressure,  
e.g. <100 mbar, and a temp. of .ltoreq.40.degree. to a concn.  
corresponding to a KHSO5 content of 25-30 wt.%, removing a partial  
stream from the conc. and cooling to <15.degree. to ppt. the triple  
salt, combining the mother liquor with the residual soln.,  
reconstituting the soln. by adding 90-100% H2SO4, 30-90% H2O2 and  
concd. KOH, and recycling to the evapn. app. Thus, 85% H2O2 6 and  
95% H2SO4 20 kg/h were continuously mixed in a flask to produce 50%  
H2SO5 contg. H2SO4, H2O, and H2O2; 0.2 m3/h of the working soln.,  
prepd. by adding 50% KOH and contg. 25-30% KHSO5 and .apprx.15%  
H2SO4 or K2SO4, is continuously removed and cooled to 5.degree. in a



1M3 crystn. flask to ppt. the triple salt. The **crystn. suspension** was removed from the flask and sepd. by centrifuge with the filtrate (.apprx.0.2 m3/h) being recycled through the recycle flask for regeneration. The filter cake was washed in the centrifuge, dried, and stored.

IC ICM C01D005-00

ICS C01B015-08

CC 49-6 (Industrial Inorganic Chemicals)

IT 37222-66-5P

(**prepn.** of, from **hydrogen peroxide**  
and potassium hydroxide and sulfuric acid)

L80 ANSWER 6 OF 6 HCA COPYRIGHT 2003 ACS on STN

82:158288 Abrasion-resistant sodium percarbonate with large particle size. Dillenburg, Helmut; Honig, Helmut; Siegel, Rudolf  
(Peroxid-Chemie G.m.b.H.). Ger. Offen. DE 2328803 19750109, 18 pp.  
(German). CODEN: GWXXBX. APPLICATION: DE 1973-2328803 19730606.

AB Abrasion-resistant, coarse grained, stable, and free-flowing Na percarbonate ( $2\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}_2$ ) is **made** by reacting a 40-60%  $\text{H}_2\text{O}_2$  soln. contg. a Mg salt with a  $\text{Na}_2\text{CO}_3$  suspension contg. 3-6 g of Na hexametaphosphate or Na polyacrylate per l. at 10-30.degree.. The  $\text{Na}_2\text{CO}_3$  suspension contains 60-100 g solid carbonate/l. in excess of the satn. value and also 190-220 g  $\text{NaCl}$ /l. Thus, 6.5 m3 of a soda suspension with  $\text{Na}_2\text{CO}_3$  260, and  $\text{NaCl}$  200, and ( $\text{NaPO}_3$ )<sub>6</sub> 4g/l. is continuously mixed over 20 min with 0.844 m3 of a 50%  $\text{H}_2\text{O}_2$  soln. contg. 70 g  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ /l. To the percarbonate suspension obtained, 10 l. water glass (36.degree.Beaume), dild. with 10 l.  $\text{H}_2\text{O}$ , are added as an active-O stabilizer. The **cryst. suspension** is centrifuged and the wet crystal mass dried at 90.degree.. The percarbonate obtained has an active O content of 13.7% and a bulk d. of 0.88 kg/l.

IC C01B

CC 49-5 (Industrial Inorganic Chemicals)

=> d 167 1-12 cbib abs hitind

L67 ANSWER 1 OF 12 HCA COPYRIGHT 2003 ACS on STN

136:264761 Preliminary results of hydrogen peroxide trials at Gledhow refinery. Davis, S. B.; Moodley, C. L.; Govender, B.; Xaba, M.  
(Sugar Milling Research Institute, University of Natal, Durban, 4041, S. Afr.). Proceedings of the Annual Congress - South African Sugar Technologists' Association, 74th, 358-360 (English) 2000.  
CODEN: PSATAA. ISSN: 0373-045X. Publisher: South African Sugar Technologists' Association.

AB Trials were undertaken at a South African sugar refinery to det. the effectiveness of  $\text{H}_2\text{O}_2$  dosing under factory conditions for color removal.  $\text{H}_2\text{O}_2$  was dosed into the raw melt at a rate of 200 ppm on brix. The  $\text{H}_2\text{O}_2$  apparently eliminated a color increase from melt to limed liquor, but the 1st and 2nd sugar colors were little changed. However, the colors of the 3rd and 4th sugars dropped significantly, resulting in a lower color for the packed refined sugar. Thus, the

use of H2O2 addn. to the raw melt was considered to be of benefit.

CC 44-3 (Industrial Carbohydrates)

ST **hydrogen peroxide** dosing raw melt

color removal sucrose manuf

IT Bleaching

(**hydrogen peroxide** dosing of raw melt  
for color removal in sucrose manuf.)

IT 57-50-1P, Sucrose, **preparation**

(**hydrogen peroxide** dosing of raw melt  
for color removal in sucrose manuf.)

IT 7722-84-1, Hydrogen peroxide, uses

(**hydrogen peroxide** dosing of raw melt  
for color removal in sucrose manuf.)

L67 ANSWER 2 OF 12 HCA COPYRIGHT 2003 ACS on STN

136:65391 NAD-dependent malate dehydrogenase protects against oxidative damage in Escherichia coli K-12 through the action of oxaloacetate.

Oh, Tae Jeong; Kim, In Gyu; Park, Seon Young; Kim, Kug Chan; Shim, Hye Won (Department of Radiation Biology, Environmental Radiation Research Group, Korea Atomic Energy Research Institute, Yusong, Taejeon, 305-600, S. Korea). Environmental Toxicology and Pharmacology, 11(1), 9-14 (English) 2002. CODEN: ETOPFR. ISSN: 1382-6689. Publisher: Elsevier Science B.V..

AB Reactive oxygen species including hydrogen peroxide (H2O2) and hydroxyl radical (.sqroot.OH) can be generated by ionizing radiation and has the potential to induce diseases. We provide the evidence that NAD-dependent malate dehydrogenase (MDH) is involved in the antioxidant role in preventing H2O2 or .gamma.-radiation-induced damage in Escherichia coli through the action of oxaloacetate. The E. coli mdh mutant strain defective in MDH activity was more sensitive to H2O2 or .gamma.-radiation than was the wild type strain, when challenged in the exponential growth phase. The mdh mutant cells pretreated with oxaloacetate (2.5 mM), a product of NAD-dependent MDH activity, prior to H2O2 treatment or .gamma.-irradn. are resistant to H2O2 or .gamma.-radiation-induced damage, so cell survivability is restored to similar levels with the wild type. The SOS induction of umu'-'lacZ **fusion** gene by H2O2 is significantly repressed by pretreatment of oxaloacetate in a dose-dependent way. These results indicate that oxaloacetate effectively protects E. coli cells against damage caused by oxidative stress. Oxaloacetate strongly prevented the DNA strand breaks by .sqroot.OH in a metal-catalyzed oxidn. (MCO) system that **generated H2O2** as a mediator. By contrast, the prevention of DNA damage by oxaloacetate in an .gamma.-irradn. system that directly generates .sqroot.OH from H2O in vitro was far less than that in an MCO system. Our results demonstrated that oxaloacetate, metabolite of NAD-dependent MDH action, plays a role as an antioxidant, possibly by scavenging H2O2.

CC 4-3 (Toxicology)

Section cross-reference(s): 8

L67 ANSWER 3 OF 12 HCA COPYRIGHT 2003 ACS on STN

116:86168 A simplified sugar refining process. Cordovez Z., Fernando; Mendoza, Juan J.; ~~Prato, Victor H.~~ (Cent. El Palmar, Aragua, Venez.). Publication of Technical Papers and Proceedings of the Annual Meeting of Sugar Industry Technologists, 50, 133-40 (English) 1991. CODEN: PTPPAC. ISSN: 0099-9032.

AB A simplified sucrose refining process currently in use in Venezuela consists of decolorization with **H2O2** and **melt** phosphatation followed by filtration through automatic deep-bed filters that require no filter aid. The substitution of powd. activated carbon with **H2O2** as a decolorant **makes** it possible to use the deep-bed filters, omitting entirely the use of filter aids, and these filters are regenerated by backwashing with filtered liquor requiring no unsweetening.

CC 44-4 (Industrial Carbohydrates)

IT 57-50-1P, Sucrose, preparation  
(manuf. of, refining in, with **hydrogen peroxide**  
decolorization and **melt** phosphatation and filtration  
through deep-bed filters requiring no filter aids)

L67 ANSWER 4 OF 12 HCA COPYRIGHT 2003 ACS on STN

~~115:144850 Electrochemical reduction of dissolved oxygen in molten amides. Sampath, S.; Narayan, R. (Dep. Chem., Indian Inst. Technol., Madras, 600 036, India). Journal of the Electrochemical Society, 138(8), 2267-72 (English) 1991. CODEN: JESQAN. ISSN: 0013-4651.~~

AB Oxygen has a high soly., 3.9 .times. 10-3M, in molten acetamide at 85.degree. and .apprx.2 .times. 10-4M in a molten amide mixt. of urea-acetamide and NH4NO3 at 25.degree.. The O undergoes only a 1-step, 2-electron, irreversible redn. at both glassy C and Pt electrodes. The electrodes are used both in the stationary and in the rotating mode. The diffusion coeffs. of O at 25-85.degree. are evaluated. The product of the redn. is peroxide, which decomp. at >50.degree., releasing more O at the interface. The rate consts. for the thermal decompn. of H2O2 are evaluated at different temps.

CC 72-2 (Electrochemistry)

Section cross-reference(s): 67

ST oxygen redn electrochem molten amide; electroredn kinetics  
**hydrogen peroxide formation**; acetamide  
urea ammonium nitrate electrolyte; disproportionation thermal  
decompn hydrogen peroxide

IT Kinetics of thermal decomposition

(of **hydrogen peroxide**, in molten

amides, electrochem. redn. of oxygen in relation to)

IT 7722-84-1P, **Hydrogen peroxide**,  
**preparation**

(**formation** of, in electrochem. redn. of oxygen in  
molten amides)

L67 ANSWER 5 OF 12 HCA COPYRIGHT 2003 ACS on STN

~~113:129279 A 25-kDa fraction from Mycobacterium tuberculosis that inhibits leukocyte bactericidal activity: reversal by gamma interferon and clofazimine. Wadee, A. A. (Sch. Pathol., Univ.~~

Witwatersrand, Johannesburg, 2000, S. Afr.). Research in Microbiology, 141(2), 249-52 (English) 1990. CODEN: RMCREW. ISSN: 0923-2508.

- AB A 25-kDa glycolipoprotein fraction from *M. tuberculosis* significantly inhibited the intracellular killing ability of cultured monocytes. When gamma interferon or clofazimine were used independently at optimal concns. (200 units/mL gamma interferon; 2 .mu.g/mL clofazimine) a significant, though not complete, reversal of intracellular killing of *Staphylococcus aureus* was obsd. Synergistic effects of the two agents, however, were obsd. at suboptimal doses, with complete restoration appearing at concns. of 25 units/mL of gamma interferon and 0.3 .mu.g/mL of clofazimine. It is noteworthy that when these agents were added to cultures prior to treatment of monocytes with the 25-kDa fraction, inhibition of intracellular killing and phagosome-lysosome fusion was not seen. However, if cultures were pretreated with the 25-kDa fraction, followed by the addn. of the two agents, no such restoration of phagocyte function was obsd. This appears to be true even when optimal concns. of gamma interferon or clofazimine were used. These and previous studies suggest that *M. tuberculosis* organisms ingested by phagocytic cells could, on degrdn., release the 25-kDa fraction which may compromise the microbicidal activity of the phagocyte by inhibiting a no. of antibacterial mechanisms. These include phagosome-lysosome **fusion**, **H2O2 prodn** ., lysozyme release and hexose monophosphate shunt activity. This could lead to the intracellular persistence of newly ingested mycobacteria, allowing them to accumulate intracellularly, as seen in leprosy and tuberculosis.
- CC 10-6 (Microbial Biochemistry)  
Section cross-reference(s): 15

- L67 ANSWER 6 OF 12 HCA COPYRIGHT 2003 ACS on STN  
110:133330 Capacity of recombinant gamma interferon to activate macrophages for Salmonella killing activity. Kagaya, Keiko; Watanabe, Koji; Fukazawa, Yoshimura (Dep. Microbiol., Yamanashi Med. Coll., Yamanashi, 409-38, Japan). Infection and Immunity, 57(2), 609-15 (English) 1989. CODEN: INFIBR. ISSN: 0019-9567.
- AB The ability of recombinant .gamma.-interferon (rIFN-.gamma.) to activate macrophages for Salmonella killing activity was kinetically examd. in relation to phagosome-lysosome **fusion** and **H2O2 generation**. Resident peritoneal macrophages of BALB/c mice incubated with 102-103 U of rIFN-.gamma./mL for 12 h exhibited enhanced bactericidal activity against *Salmonella typhimurium*, although **H2O2 generation** was unaltered. In contrast, macrophages incubated with equal doses of rIFN-.gamma. for 48 h showed both an enhanced *Salmonella* killing activity and an increased **H2O2 generation**. Thus, rIFN-.gamma. per se can activate peritoneal macrophages to induce *Salmonella* killing activity and apparently increased phagosome-lysosome fusion followed by an O-independent killing mechanism is primarily responsible for the enhanced *Salmonella* killing activity in rIFN-.gamma.-activated macrophages.

CC 15-5 (Immunochemistry)

IT 7722-84-1, **Hydrogen peroxide**, biological studies  
(**formation** of, by interferon-activated macrophage in  
Salmonella killing)

L67 ANSWER 7 OF 12 HCA COPYRIGHT 2003 ACS on STN

110:68736 Photometric determination of total rare earths in wolframite  
using Arsenazo-DBS. Wu, Fengxiang (Anal. Cent., Jiangxi Prov. Bur.  
Geol. Min., Peop. Rep. China). Yankuang Ceshi, 7(1), 21-3 (Chinese)  
1988. CODEN: YACEEK. ISSN: 0254-5357.

AB Wolframite sample was fused with Na<sub>2</sub>O<sub>2</sub>; tungstic acid was sepd. as a  
ppt. by heating the HCl soln. (contg. **H<sub>2</sub>O<sub>2</sub>**) of the  
**fusion products**. Rare earths were preconcd. by  
liq. chromatog. on CL-PMBP Levextrel resin and were eluted with 0.5%  
HCl. The total rare earth was detd. by measuring the absorbance at  
630 nm with Arsenazo-DBS as color reagent in the medium contg. 0.18M  
HCl, EDTA and ascorbic acid.

CC 79-6 (Inorganic Analytical Chemistry)

L67 ANSWER 8 OF 12 HCA COPYRIGHT 2003 ACS on STN

93:116616 Lightweight oxygen source. Ando, Eisaku (Japan). Jpn. Kokai  
Tokkyo Koho JP 55042237 19800325 Showa, 4 pp. (Japanese). CODEN:  
JKXXAF. APPLICATION: JP 1978-114263 19780918.

AB A simple lightwt. source of O<sub>2</sub> for an emergency breathing device is  
provided by coating a catalyst for decompn. an aq. H<sub>2</sub>O<sub>2</sub> soln. with a  
substance that is not dissolved in H<sub>2</sub>O at ambient temp. but that  
does melt at 50-120.degree. so that O<sub>2</sub> is gradually generated. The  
lower part of the rod-shaped hopcalite catalyst is coated with a  
polymer, m. 50-120.degree., and paraffin so that when the catalyst  
is dropped into the aq. H<sub>2</sub>O<sub>2</sub> reaction occurs only in the upper zone  
of the soln. The layer of soln. surrounding the catalyst is heated  
by the reaction to melt the coating and uncover more of the catalyst  
and gradually the reaction zone is lowered and the rate of O<sub>2</sub>  
generation is const. for several min.

IC C01B013-02; A62B007-08

CC 49-1 (Industrial Inorganic Chemicals)

IT Coating materials

(paraffin-polymer, water-insol. low-melting, for  
**hydrogen peroxide** decompn. catalysts)

IT 7782-44-7P, **preparation**

(**generation** of, by **hydrogen peroxide**  
decompn., coating with catalyst with water-insol. low-melting  
material for)

L67 ANSWER 9 OF 12 HCA COPYRIGHT 2003 ACS on STN

85:18004 Physiology and ultrastructure of the blood platelet following  
exposure to hydrogen peroxide. Redvien, Robert, Lindon, Jack N.;  
Levine, Peter H. (Blood Coag. Lab., New England Med. Cent. Hosp.,  
Boston, MA, USA). British Journal of Haematology, 33(1), 19-26  
(English) 1976. CODEN: BJHEAL. ISSN: 0007-1048.

AB H<sub>2</sub>O<sub>2</sub> in micromolar concns. induced shape changes in human blood  
platelets, and modified the aggregation and release reaction of

these cells as induced by ADP or thrombin. In larger (millimolar) concns., **H2O2 produced fusion** of platelets with distortions in platelet morphol. unlike those normally caused by aggregating agents. Thus, the **prodn.** of **H2O2** in vivo by granulocytes or other cells could influence the processes of hemostasis or thrombosis.

CC 13-5 (Mammalian Biochemistry)

L67 ANSWER 10 OF 12 HCA COPYRIGHT 2003 ACS on STN

74:115801 2-Imidazoline derivatives as developers for hair dyes. Lange, Fritz W.; Mueller, Jens E. (Schwarzkopf, Hans, G.m.b.H.). Ger. Offen. DE 1941100 19710225, 20 pp. (German). CODEN: GWXXBX. APPLICATION: DE 1969-1941100 19690813.

GI For diagram(s), see printed CA Issue.

AB The title compds. (I) were used in H2O2-contg. developer solns. for hair dyes. Thus, an opaque developer soln. contg. 6% H2O2 was prepd. by melting 20 g I (R = C11H23, R1 = NH2) at 80.degree., addn. of 689 g H2O of 75.degree., cooling to 20.degree., and addn. of 91 ml 8.5% H3PO4 to adjust to pH 4.2 and 200 ml 30% H2O2. Also used were, e.g., I where R1 = OH and R = Me(CH2)10, Me(CH2)16, or Me(CH2)7CH:CH(CH2)7.

IC A61K

CC 62 (Essential Oils and Cosmetics)

L67 ANSWER 11 OF 12 HCA COPYRIGHT 2003 ACS on STN

39:25200 Original Reference No. 39:4018c-i,4019a Quantitative microchemical analysis of minerals, ores, and rocks. VI.

Determination of chromium and vanadium. Frid, B. I. Zavodskaya Laboratoriya, 11(No. 1), 17-23 (Unavailable) 1945. CODEN: ZVDLAU. ISSN: 0321-4265.

AB The colorimetric method proposed for detg. Cr is based on the formation of a violet color from the reaction of CrO4-- with diphenylcarbazide in acid soln. The volumetric method for the detn. of Cr and V is based on the modified micromethod of Syrokomskii and Stepin. (C.A. 30, 4780.8). For colorimetric detn. of Cr fuse the 10-20-mg. sample with 0.5 g. of Na2CO3 in a 5-6-ml. Pt crucible, ext. the melt with hot water, filter into a conic 50-ml. flask through a microfiltering tube filled with macerated paper, and wash 5-6 times with a hot 1% Na2CO3 soln. in 0.5-ml. portions. Add to the alk. soln. H2SO4 (1:2) until the yellow color of paranitrophenol disappears, shake until most of the CO2 is evolved, add 1 ml. of 2% AgNO3 and 0.2 g. of (NH4)2S2O8, cover with a watch glass, boil for 3 min., cool, and decomp. HMnO4 by adding several grains of NaN3 (avoiding excess). Transfer the soln. to a 25-ml. cylinder or measuring flask, add 1 ml. of 6N H2SO4 and 1 ml. of 0.5% diphenylcarbazide, add water to 25 ml., mix carefully, and match the colored soln. with a standard soln. (0.0005-0.0002 N K2Cr2O7) in a microcolorimeter. A photcolorimeter can be used for slightly colored solns. For the volumetric detn. of Cr fuse 10-20 mg. of the sample with 1 g. of K2S2O8 in a porcelain crucible, treat the melt with 0.5 ml. of H2SO4 (1:1) and boil until dissolved. To 5-6 ml. of hot soln. add 1 ml. of 2% AgNO3 and 0.2 g. of (NH4)2S2O8 and

continue as usual, titrating with  $\text{FeSO}_4$  soln. in the presence of phenylanthranilic acid as indicator. The indicator is prepd. by dissolving 0.05 g. of phenylanthranilic acid in 100 ml. of water contg. 0.1 g. of  $\text{Na}_2\text{CO}_3$ . For volumetric detn. of V fuse 10-30 mg. of the sample in a 5-6-ml. Pt crucible with 0.5 g. of  $\text{Na}_2\text{CO}_3$  to which several grains of  $\text{Na}_2\text{O}_2$  have been added, ext. the melt with water, transfer to a 50-ml. Erlenmeyer flask, neutralize with  $\text{H}_2\text{SO}_4$  (1:1), using several drops of excess, and decomp. the **H<sub>2</sub>O<sub>2</sub> formed during fusing** by boiling or by adding  $\text{KMnO}_4$ . In the presence of large quantities of Fe (titanomagnetite) not all the V from the alk. ext. is obtained by a single fusion; up to 25% of V remains in the residue. Therefore, for more accurate results, fuse the sample a 2nd time and combine the exts. Oxidized ores can be fused with  $\text{K}_2\text{S}_2\text{O}_8$  in a porcelain crucible. In such cases ext. the melt with water, add 2-3 ml. of  $\text{H}_2\text{SO}_4$ , reduce V and Cr with excess 0.01 N Mohr's soln., add first 1 ml. of  $\text{H}_3\text{PO}_4$  (1:1), then carefully in the cold 0.01 N  $\text{KMnO}_4$  until the pale pink color persists for 3 min., decomp. the excess  $\text{KMnO}_4$  by adding dropwise 0.2%  $\text{NaNO}_2$  (avoiding excess  $\text{NO}_2^-$ ), and add immediately 0.2 g. of urea. The vol. of the final soln. contg. little V should not exceed 3-5 ml. and that contg. greater quantities of V should not exceed 10 ml. To this soln. add after 5 min. 3-5 ml. of  $\text{H}_2\text{SO}_4$  (1:1) and 1 drop of 0.5% phenylanthranilic soln., and titrate with 0.001-0.01 N Mohr's salt soln. (depending on the content of V) until the color changes from violet to pale green. The end point is very sharp. 1 ml. of 0.01 N Mohr's salt soln. corresponds to 0.9143 mg. of  $\text{V}_2\text{O}_5$ . Fifteen references.

CC 7 (Analytical Chemistry)

L67 ANSWER 12 OF 12 HCA COPYRIGHT 2003 ACS on STN

29:26274 Original Reference No. 29:3420d-g Plastic compositions.

Twiss, Douglas F.; Neale, E. T. (Dunlop Rubber Co. Ltd.). GB 420386 19341130 (Unavailable). APPLICATION: GB .

AB Plastic compns. of org polysulfide plastics, of the kind obtained by the reaction of halogenated hydrocarbons with a polysulfide or thiosulfate or an alkali or alk. earth metal, and synthetic resins of the PhOH-aldehyde, polyhydric alc.-polycarboxylic acid and aldehyde-arylamine types are made by dissolving the org. polysulfide plastic in 1 of the parent substances for the resin and completing the formation of the resin after the addn. of the remaining parent substance(s). The products are suitable for addn. to rubber for improving its impermeability and oil- and solvent-resistance. Among examples, (1) diethylene tetrasulfide (I) (obtained by refluxing a mixt. of an alc. soln. of ethylene dichloride and aq.  $\text{Na}_2\text{S}_2\text{O}_3$  and heating the **product** with **H<sub>2</sub>O<sub>2</sub>**) is dissolved in **molten** PhOH and the soln. is treated with formalin in the presence of  $\text{HCl}$ ; the product may be added to a rubber mix contg. S,  $\text{ZnO}$ , mercaptobenzothiazole, stearic acid and C black, which is then vulcanized, (2) I is dissolved in hot  $\text{PhNH}_2$  and the mixt. is refluxed with aq.  $\text{CH}_2\text{O}$ .

CC 13 (Chemical Industry and Miscellaneous Industrial Products)